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# JOURNAL

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## Cellulose Acetate Toy Automobiles

—Molded by Aluminum Model Toys of Detroit

*(Photo courtesy Eastman Chemical)*

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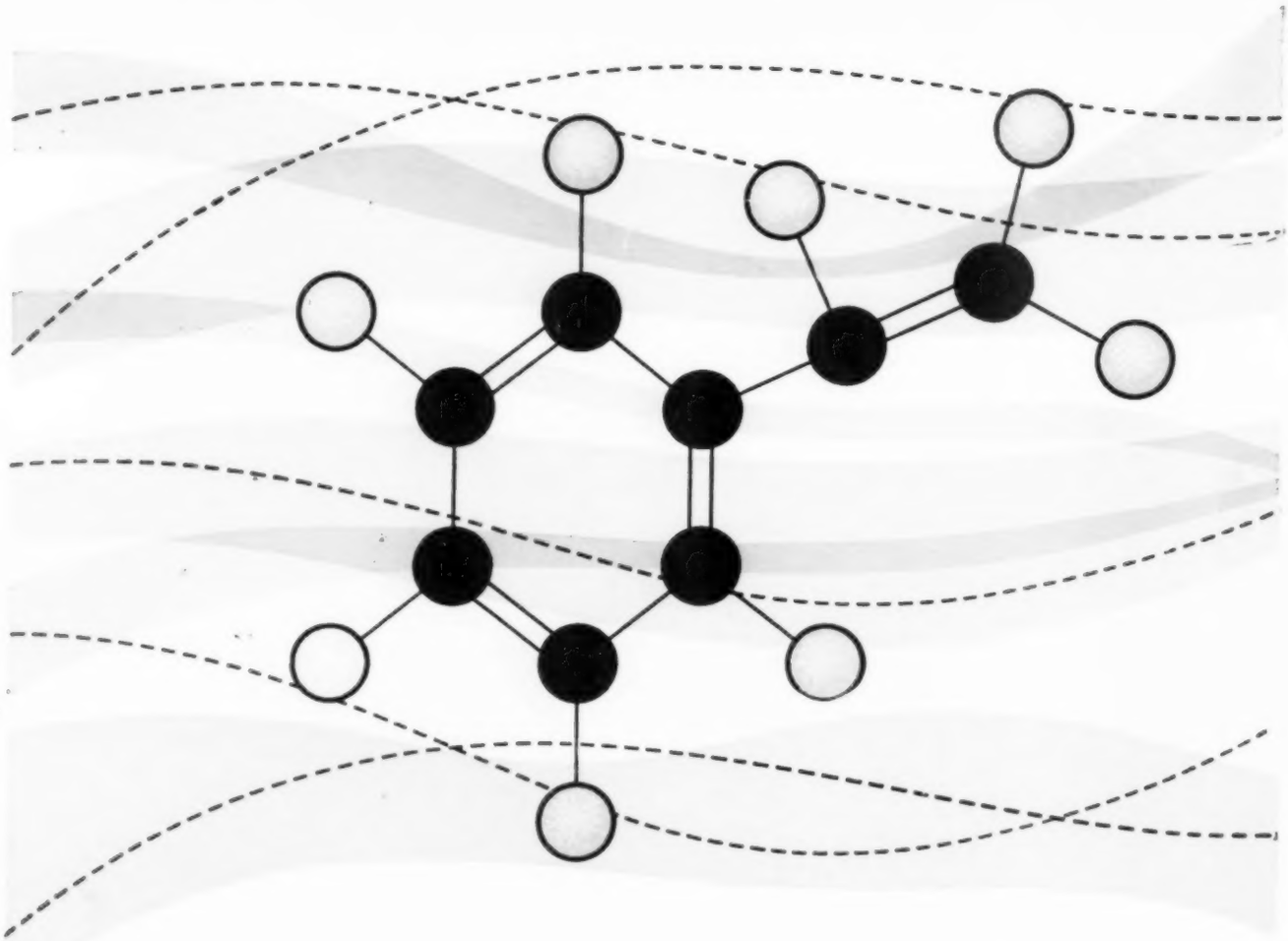
# STYRENE



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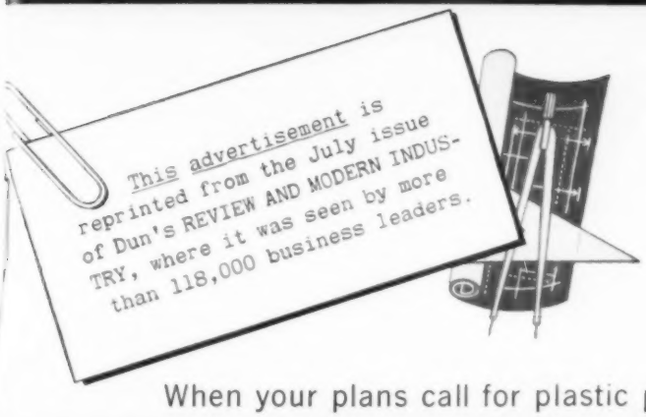
# Craftsmen of the 20th Century

No. 17 of a series to introduce you to some of industry's outstanding plastics craftsmen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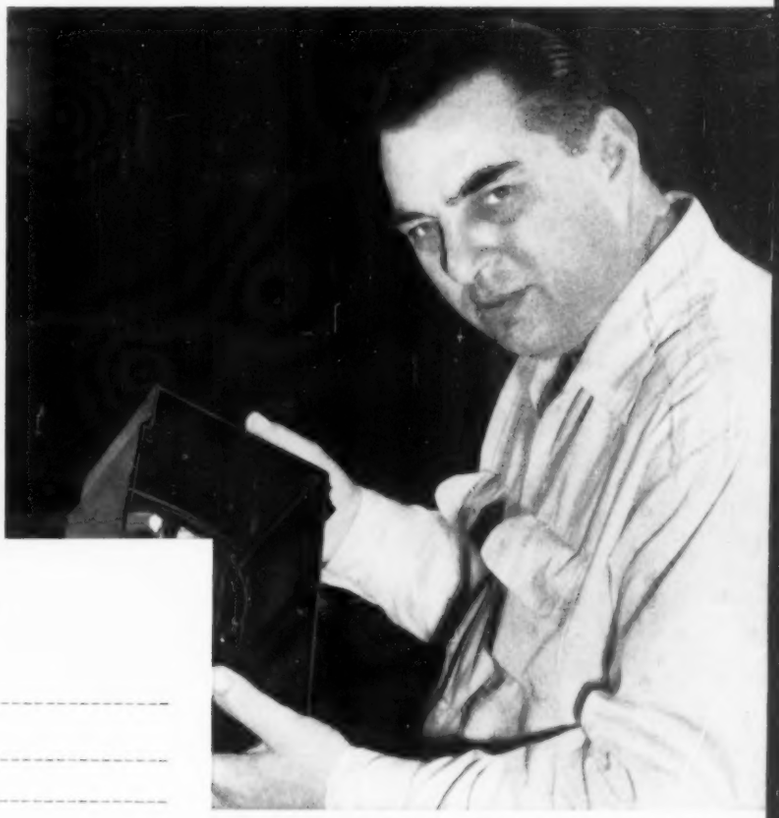
Today, a fabulous new family of materials is channeling talents in new directions. Modern craftsmen are designing products in versatile plastics—and mass-producing them at reasonable cost.

Pictured here are two of the specialists who are creating plastic products that are serving every industry, every home.

Monsanto, a major producer of high-quality plastic materials, salutes these craftsmen who are helping to mold America's tomorrow.

When your plans call for plastic parts or products, consult an expert custom molder



**Molded Products Division, Admiral Corp., West Chi-**
our brothers, Mr. Szorc has been an eye-witness to
ustom molding since 32 years ago, when he oper-
press turning out ladies' compacts. Now superin-
sion molding at the huge new Admiral plant, Mr.
e operation of 27 semi-automatic hydraulic presses
on press as tall as a 3-story building where 4 TV
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ackground and experience in the molding industry,
ut, "Compression molding has made tremendous
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mplexity of jobs handled. With the opportunities
vide for reducing the number of components by
rts in a single unit, it is possible, for example, to
radio speaker in less than 2 minutes."

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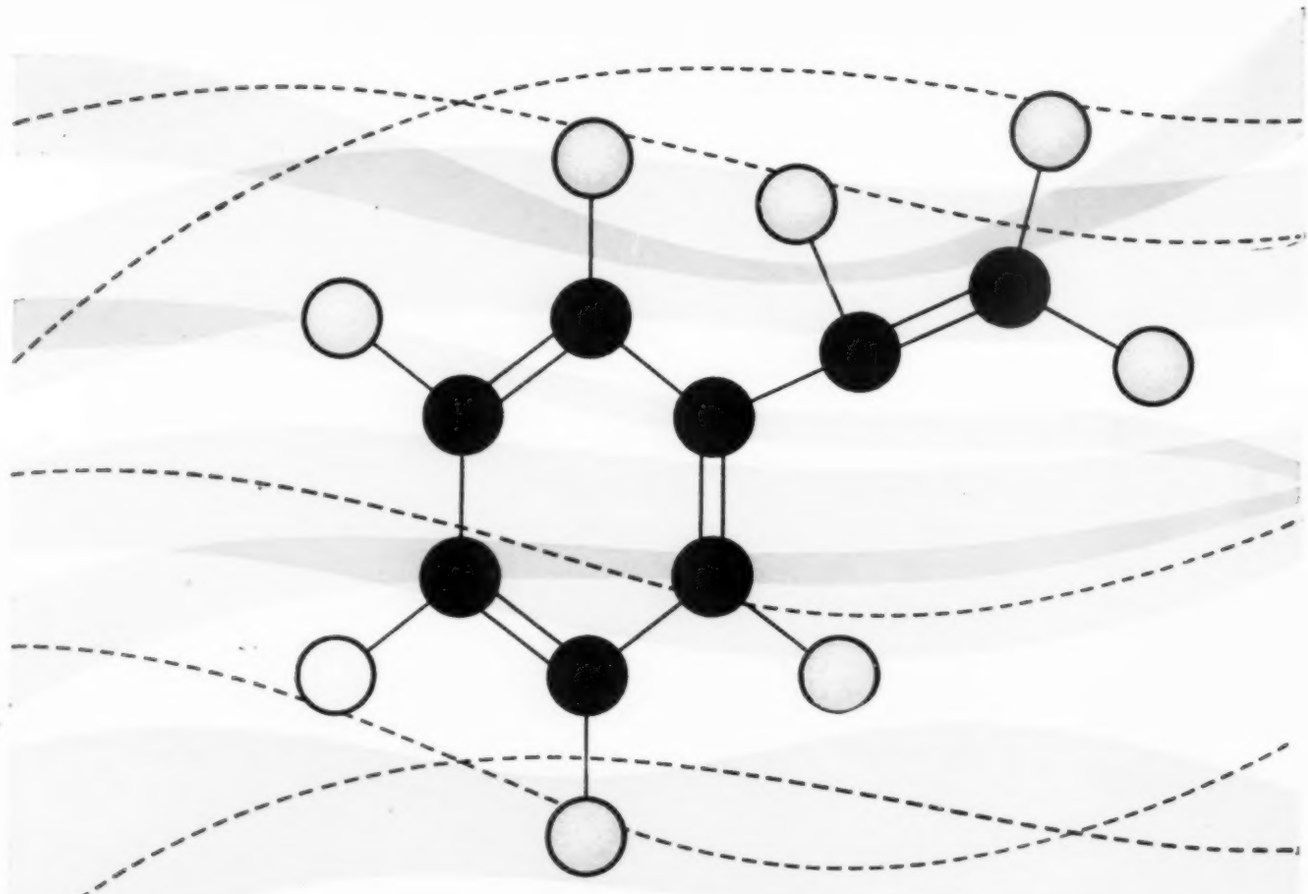

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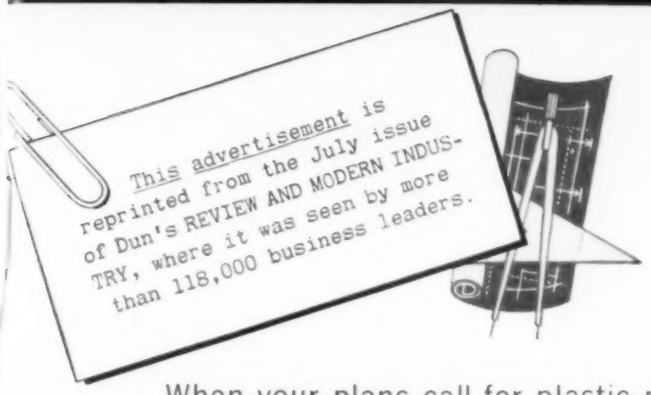
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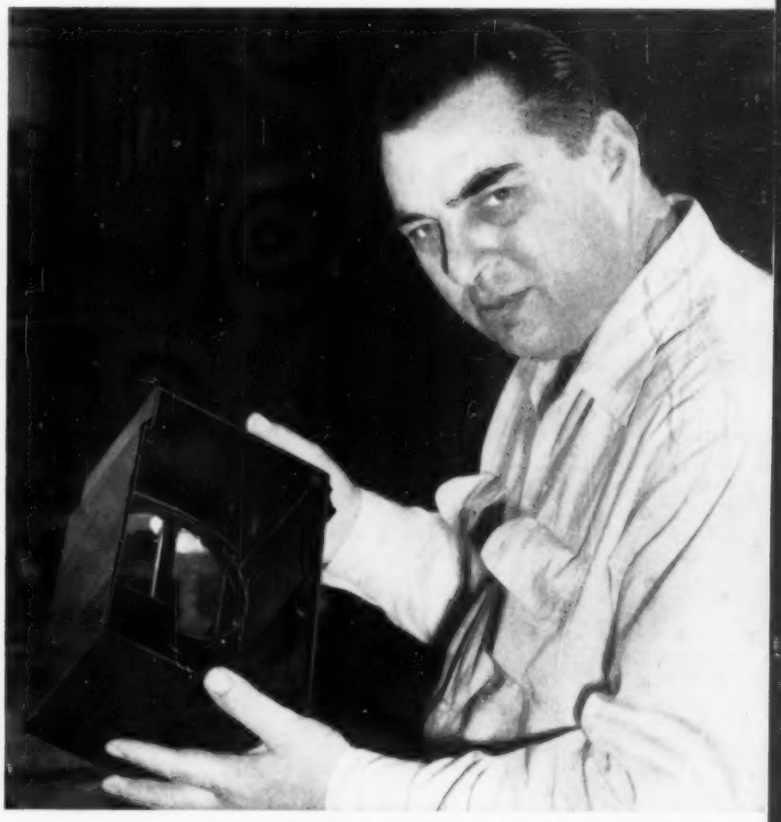
## When your plans call for plastic parts or products, consult an expert custom molder

**Clyde E. Morrell, Proion Plastics Company, Florence, Mass.** Fifteen years ago Clyde Morrell took a job as a draftsman in a newly formed plastics operation. Today, at 37, he is recognized as a top man in his field—Chief Mold Designer for one of the largest custom molding companies in America. Mr. Morrell is the man to whom manufacturers put their questions: What will a mold for my product cost? Will it satisfy my production requirements? Is a redesign of my product necessary to guarantee trouble-free production? Under his supervision the mold design department engineers the custom mold, expedites its construction and assumes responsibility for its successful operation.

In spite of Mr. Morrell's long experience, he says, "It's a young industry. We are constantly developing new ideas and techniques. To this end our professional association, The Society of Plastics Engineers, is earnestly devoted. I consider myself fortunate to have grown with an industry that has such a limitless future."



**Stephen J. Szorc, Molded Products Division, Admiral Corp., West Chicago, Ill.** Like his four brothers, Mr. Szorc has been an eye-witness to many changes in custom molding since 32 years ago, when he operated a small hand press turning out ladies' compacts. Now superintendent of compression molding at the huge new Admiral plant, Mr. Szorc supervises the operation of 27 semi-automatic hydraulic presses including a 3,000 ton press as tall as a 3-story building where 4 TV cabinets can be made at the same time.

From his wide background and experience in the molding industry, Mr. Szorc points out, "Compression molding has made tremendous strides in the past 10 years, not only in the size of equipment being used, but in the complexity of jobs handled. With the opportunities which plastics provide for reducing the number of components by molding several parts in a single unit, it is possible, for example, to produce a complete radio speaker in less than 2 minutes."

MONSANTO CHEMICAL COMPANY,
PLASTICS DIVISION, SPRINGFIELD 2, MASS.

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PHENOLIC MOLDING COMPOUNDS

# U.S.I. POLYETHYLENE PROCESSING TIPS

Vol. 1, No. 4

## Molders Can Minimize Shrinkage

Shrinkage of injection molded items is often a troublesome problem for molders. In closures, and in components which are to become part of an assembly, excessive shinkage can be a major cause of rejects. The growing use of polyethylene for such parts has sparked molder interest in shrinkage control on items made from polyethylene resins.

While a certain amount of shrinkage is inherent in any process that involves cooling of a material from elevated temperatures, it is possible for injection molders to minimize the condition to acceptable levels.

In polyethylene, such control may be achieved by the right choice of operating conditions and by selection of a resin with characteristics to fit them.

## Lower Temperatures Key to Less Shrinkage

Since shrinkage is a natural result of cooling, a main avenue of attack on the problem is to mold at lower temperatures. Here are some procedures that tend to reduce shrinkage:

***Reduce barrel temperature and run a cold mold.***

***Use high pressure and long inject time*** to pack the mold as much as possible, short of causing the item to stick.

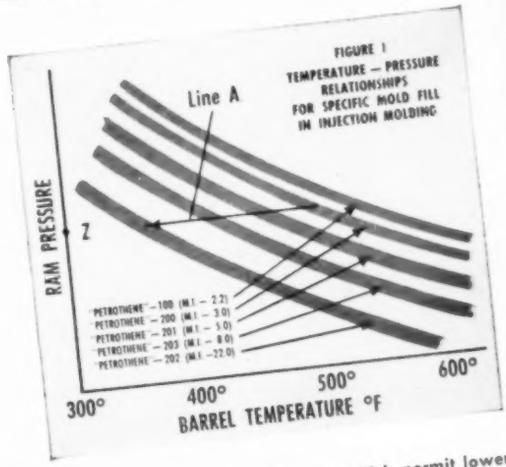
***Allow longer dwell time.*** This helps to equalize temperatures throughout the mass so that cooling takes place at a uniform rate.

This point deserves further comment. Since the last material to flow into the mold is considerably hotter than the plastic at the extremity, a substantial temperature differential may still exist when the mold is opened. This uneven cooling results in non-uniform shrinkage which, in turn, causes the item to warp. Longer dwell time permits some equalization of this temperature difference and thereby keeps uniform the shrinkage that does occur. (The problem of warpage is discussed more fully in a previous issue of "Processing Tips". Write for a reprint if you missed it.)

## High Melt Index Resins For Lower Molding Temperatures

Polyethylene resins with high melt indices can be a big factor in reducing shrinkage. In the first place, high melt index permits lower molding temperatures. This means less cooling, hence less shrinkage. At the same time, their higher flow characteristics permit tighter packing of the mold.

PETROTHENE 202 and 203 polyethylene resins are well suited to operating conditions for minimum shrinkage. The high melt indices of these resins permit reduction of injection temperatures by 70° to 130° for a given fill, cycle time and ram pressure. This is graphically illustrated by a set of curves for a particular mold:




**Curves show how resins with high M.I. permit lower molding temperatures for a given pressure and fill. At pressure Z, a resin with comparatively low M.I., such as PETROTHENE 200, may require a barrel temperature of 500°F. Under the same conditions, PETROTHENE 203 requires 430°, PETROTHENE 202 only 370°.**

## Special Problem? Call on U.S.I.

In general, the suggestions outlined here will reduce shrinkage of molded polyethylene items. Some experimentation may be necessary, of course, to achieve a satisfactory result for your particular situation. In many cases, a switch to high-flow resins such as PETROTHENE 202 or 203, where these resins are satisfactory for the end-use, will permit operation at best conditions for minimum warp.

If the nature of your product creates an unusual problem, don't hesitate to call on U.S.I. for technical assistance. Well qualified Technical Service personnel stand ready to help you find a practical and economical solution.

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# SPE JOURNAL

**Vol. 12 August, 1956 No. 8**

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The four tanks shown in this picture are part of a new line of corrosion resistant polyester glass reinforced tanks being offered by Haveg Industries, Inc. of Wilmington, Delaware. Said to have corrosion resistance to oxidizing acids and many chemicals far superior to stainless steel, these tanks are extremely light in weight. The heaviest of those in the picture weighs only 150 lbs., holds 600 gallons, can be readily put in place by two men.

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**BAKELITE Brand Phenolic** was chosen for this automatic washer soap disperser to resist impact, moisture, and chemicals, particularly mild alkaline and acid solutions.

**BAKELITE Brand Impact Styrene,** used for these molded slide viewers, provides fine gloss, rich colors. Molded latch permits finger-pressure opening of base to change batteries.

**BAKELITE Brand C-11 Plastic,** molded into "Carpetmates" furniture supports, demonstrates high flexural strength, low distortion, even when used with the heaviest furniture.

**BAKELITE Brand Impact Styrene** makes up the deck and hull of this youngsters' 10 lb. "Pirateer" skiff. Each is an extruded sheet, vacuum-formed with a planking effect.

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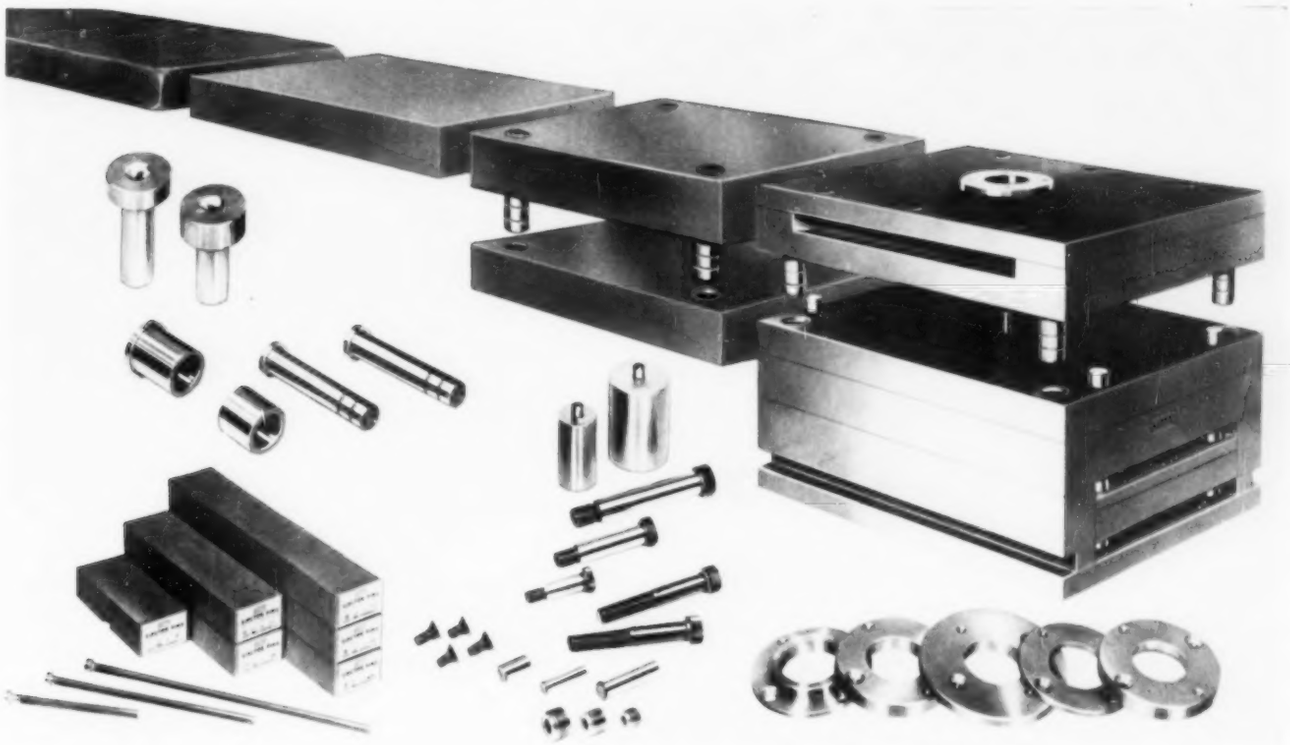

# PROGRESS THROUGH STANDARDS

## . . . and how DME "helped"

The history of economy in modern business has been directly related to the history of Standardization. One of the major elements affecting the economic production of finished plastic parts is the high initial cost of the mold. A progressive step in reducing mold cost, without sacrificing the high quality required, has been successfully achieved through Standardization.

When D-M-E originated Standard Mold Bases and their component parts in 1942, it provided the answer to the rapidly growing demand for high quality molds that would maintain and promote the economic advantages of plastic parts.

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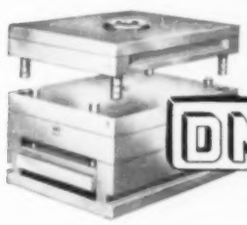


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Francis Blod, Design Associates, New York City

Mr. Blod discusses the handsome decanter that won 1st Prize, Class II, in the Koppers 1956 Design Competition with associate George Stehl. The prize-winning decanter is being marketed by Hutzler Mfg. Co., Long Island City, N. Y.

# *Designer Francis Blod talks plastics:*

"This container *had* to be made with polyethylene . . . because it offers so much to the user. It has a soft translucency . . . a graciousness that any housewife will appreciate. It does not rattle and it will be less cold to the touch. This container is safe for children . . . possesses lightness, a cover that won't come off and a non-drip spout. Too much cannot be said for the eye-appeal we get from polyethylene-designed items. We use it whenever possible in designing something that is to appeal to women or children.

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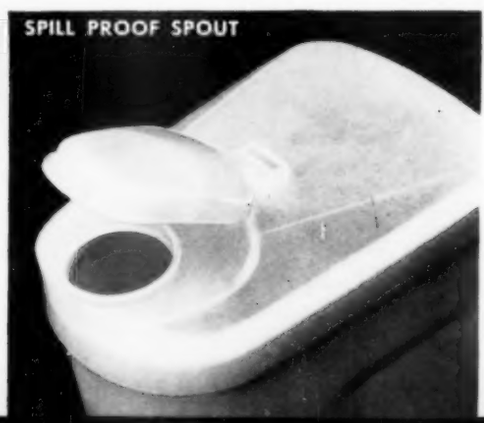

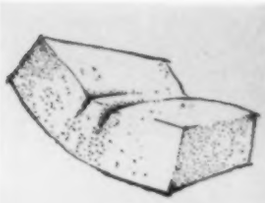

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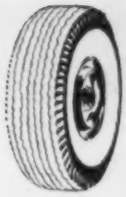

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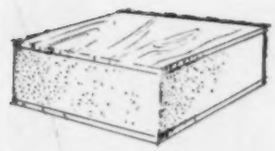

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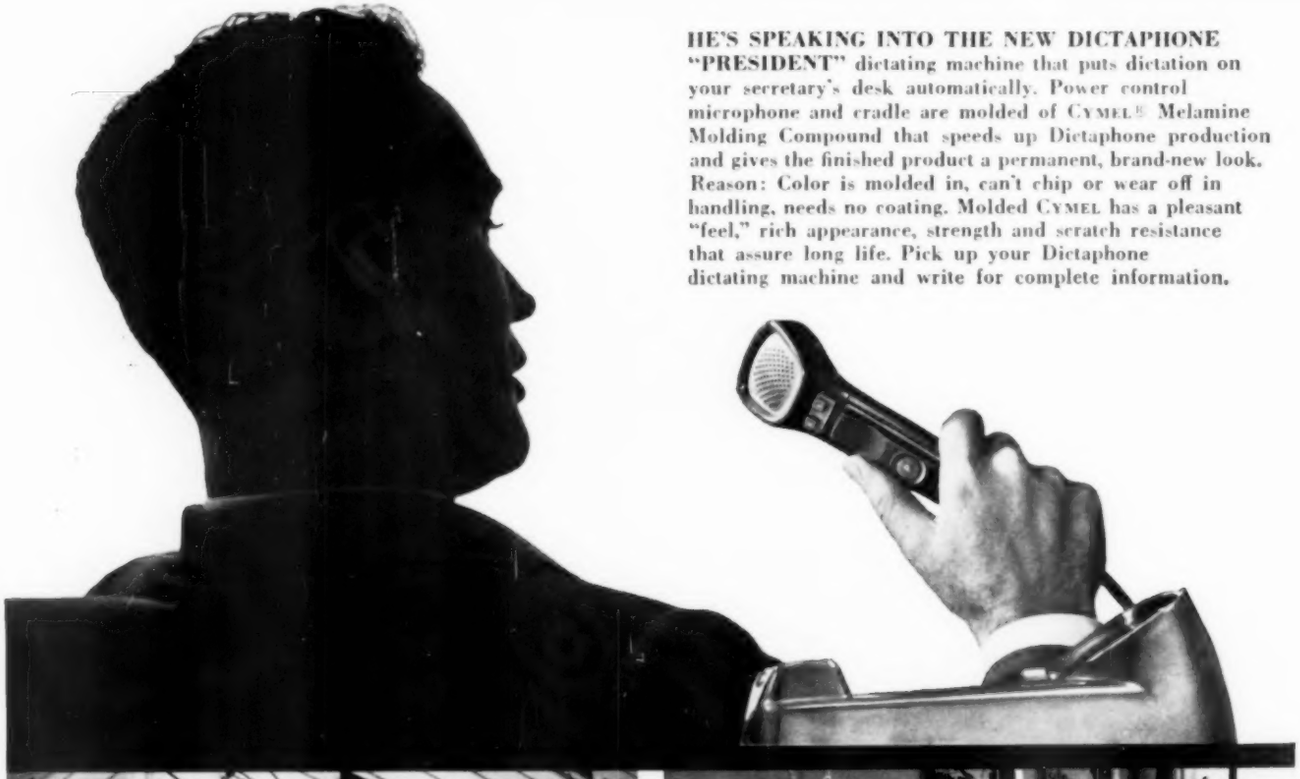

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# Reinforced Plastic Springs

F. W. Reinhart and Sanford B. Newman

*National Bureau of Standards*

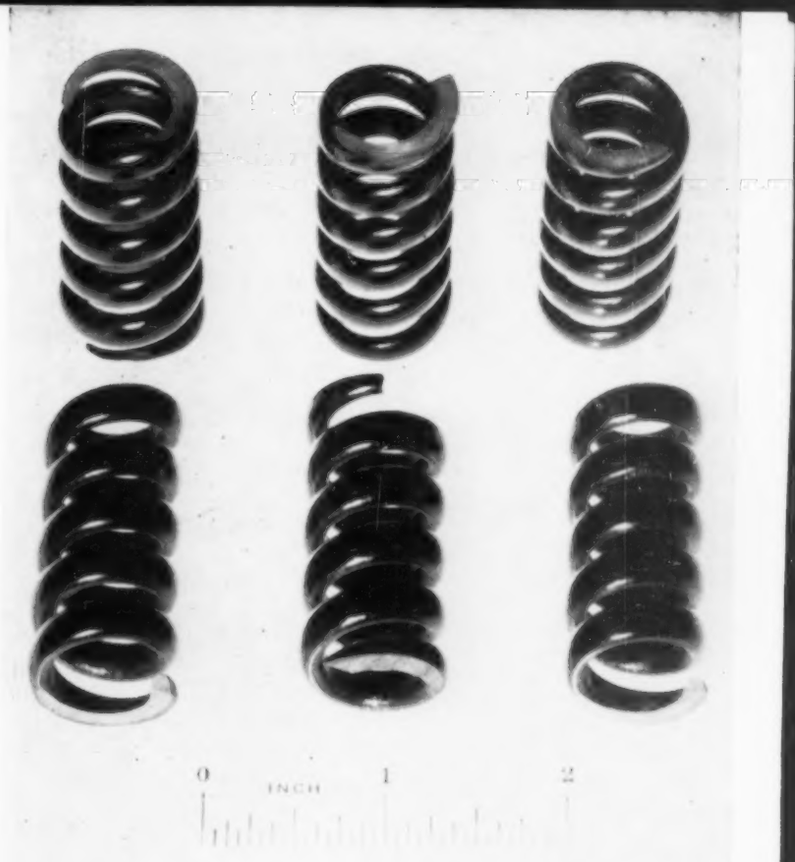


FIGURE 1. PLASTIC SPRINGS OF GLASS REINFORCED EPOXY RESIN CURED WITH 15% m-PHENYLENEDIAMINE

THE PRODUCTION OF springs requires not only a knowledge of the physical characteristics of the material used but an accurately controlled fabricating technique as well. In general, available information deals principally with steel springs or copper and copper alloy springs. The use of non-metallic materials, such as plastics or rubbers, for spring making is relatively uncommon. Polymeric substances, however, have properties advantageous to their use in springs for special purposes. For example, they are non-magnetic and have low electrical and thermal conductivity and high corrosion resistance. Some applications may benefit from the strength to weight ratios of plastics, which are often higher than those obtained with spring making metals. With their range of transparent and colored materials, striking decorative effects are also possible.

For general purposes mechanical springs may be classified into two divisions: wire springs and flat springs. These large groups can be further categorized. For example, wire springs include compression, extension, and torsion types of helical or spiral form. In its broadest sense the flat spring classification includes cantilever and elliptical strip springs, spring washers, Belleville springs, and power springs of the motor or clock type. Each of the smaller groups present individual problems to the designer and fabricator. While plastics or other non-metallics can be formed into any of these structures, the work to be described in this paper deals mainly with the production of helical springs used in compression. Some consideration was also given to the development of block compression springs for use with higher loads.

Helical springs of metal are formed by winding on mandrels. At normal temperatures, permanent set and elastic deformation both occur and as a result the spring uncoils slightly, increases in diameter, and may increase in length when removed from the mandrel. This forming also is responsible for considerable residual stress in the finished product. Plastic springs, however, may be molded directly to dimension without the development of any considerable internal stress.

Neither plastics nor metal can approximate the ideal spring and for much the same reasons. The physical properties of both groups of materials are time and temperature dependent. Plastics, however, are more sensitive to these variables than the metals commonly found in springs. Hysteresis is another phenomenon that cannot be overlooked in evaluating spring making materials. The effect of most of these factors on spring performance was studied in the springs developed during this investigation.

*The results described in this paper were obtained on a project sponsored by the Ordnance Corps, Department of the Army. The help and encouragement of Thomas E. Bartlett, R. G. Thresher, Joseph Messineo and Thomas B. Blevins of the Army Ordnance Corps and the assistance of Murray C. Slone, Desmond A. George, Leon Horn, George G. Richey, Catherine B. Simpson and George S. Saines of the National Bureau of Standards with the experimental work are gratefully acknowledged.*

*This article was presented at the SPI Reinforced Plastics meeting in February, 1956. Reprinted by permission from Product Engineering, June, 1956.* *Copyright 1956 by McGraw-Hill Pub. Co.*

## Specification for Springs

A detailed spring specification may include requirements for material, wire size, spring diameter, free length, number of coils, helix of coil, solid length, loads, type of ends and even the scale per inch. Considerable latitude was permitted, however, in developing the plastic springs to be described in this report. The principal characteristics considered during their development were the following: That the springs

1. Be manufactured from any non-metallic material except wood.
2. Be so designed that they may be easily fabricated by mass production methods.
3. Be capable of functioning at any air temperatures from −40°F to +135°F.
4. Be no larger than 2-inches in diameter and 3-inches in height.
5. Support a load of 25 ± 5 lb. at a deflection of 0.25 to 0.50 inches.
6. Immediately upon release from the loaded and/or stressed position produce approximately 30 in.-oz of kinetic energy.

## Materials and Methods of Forming

In the early stages of this work a few springs were manufactured by turning tubes of polystyrene, cellulose nitrate and paper filled phenolic on a screw cutting lathe. The lathe tool cut through the wall until only a spiral of the plastic remained. While spring-like structures were fabricated from this technique, it did not lend itself to inexpensive mass production. In addition, the final dimensions of the spirals were not always predictable because of residual stresses in the tubes. Attempts were made to produce usable springs by wrapping resin-soaked glass fiber braid or glass fiber rovings around mandrels and curing the resin in situ. This method appears to have limited usefulness.

The most successful procedure was found to be the polymerization of the spring composition in vinyl chloride-vinyl acetate copolymer tubing. Lengths of glass rovings were formed into a loose, untwisted yarn. The yarn was then doubled to form a U-shaped bundle. This bundle was placed in a trough partly filled with liquid resin and immersed until it was thoroughly soaked. A steel wire was threaded through a length of commercial vinyl copolymer tubing having a ¼-or-3/16-inch inside diameter and a ⅛-inch wall thickness. The leading end of the wire was attached to the bend in the U-shaped bundle of resin-soaked rovings and the wire was then retracted. Considerable force was applied to the wire in order to draw the impregnated filaments into the tubing. When the tubing was completely filled, the wire was disconnected and the ends sealed with hose clamps. Once the tubing was sealed, it was wound in a helix on a mandrel of suitable diameter. Tubing ends were restrained just sufficiently to hold the spiral in position. Curing of the resins was carried out under the conditions specified by the manufacturers. The entire assembly was placed in an air-circulating oven at the recommended temperature. After removal from the oven the springs were allowed to cool and the tubing removed. Post curing was performed as required. Finally, the springs were cut and ground to the finished length. Some batches of the vinyl copolymer adhered to, or fused with the resins used in forming the springs. This difficulty was corrected by first heating the tubing at 120°C for two hours or until it began to discolor.

The most promising resins used in the helical springs were epoxides and polyesters although many others were also investigated. Commercial catalysts and curing agents recommended by the resin suppliers were used in curing the springs and rods. Most of the results reported were obtained with a glass roving that had a chrome finish.

## Screening Tests for Resins

Experience with numerous resins in the course of this work has shown that the variation in the modulus of rigidity with temperature is primarily a function of the resin and, within limits, independent of the amount of glass reinforcement. Since most of the stresses in a helically wound spring are torsional, this dependence makes possible the use of a simple torsion test on rod-shaped specimens as a means for screening potential spring-making resins. The resin was simply polymerized in tubing to produce specimen rods, thus avoiding the more detailed procedure of making a complete spring.

The torsional characteristics of a number of resins could be compared in a qualitative fashion using a rod about fourteen inches long. One end of the rod was clamped rigidly in a grip and an external torque was then applied ten inches from the grip. The angle of twist under load was determined. Applied torques did not induce stresses higher than the working stresses encountered in springs. The torsional coefficient of shear or **modulus of rigidity G,** may be determined from these data. It is taken as (1)

$$G = 32\,T\,L / \pi\,\theta\,D^4 \qquad (1)$$

where T is the twisting moment (in.-lb), L is the length of the specimen rod (in.), $\theta$ is the angle of twist (rad) and D the diameter of the rod (in.) While the data for individual rods show some scatter, the average values are sufficiently reproducible to allow comparisons between resins. The rods were all tested at three temperatures, −40°F, 73°F and 135°F. The rods and torsion tester were placed in an air-circulating oven or cold box for eight to twelve hours to assure thermal equilibrium at the desired temperatures. In some cases the modulus of rigidity at 135°F was greater than the modulus determined at 73°F. This does not truly reflect the physical properties of the resin as affected by temperature but is an indication of continued cure at the higher temperature.

Results of tests of rods in Table 1 show the greater sensitivity to higher temperature of most of the polyester compositions as compared to the better epoxide compounds. Apparently, all epoxy resins will not yield the same results. No significant effect can be attributed to changes in the percentage of curing agent. It would appear that each resin will require a separate study before optimum conditions of preparation can be ascertained. As might be expected plasticizers increase the permanent set of the nonreinforced rods at high temperatures.

Plastic springs made from polyester resins with glass fiber reinforcement have shown good energy recovery properties after short periods of storage at −40°F, but these properties are adversely affected by thirteen days exposure to 135°F while under stress.

Sharp and Maxwell (2) have recently determined the modulus of rigidity of some typical plastics. The results appear in Table 2. In their group of materials, polystyrene approached the epoxies and acceptable polyesters in torsional moduli in the temperature range where comparison is possible. However, even polystyrene does not yield values of G equal to those obtained with some epoxy compositions or even those of the better polyesters. It

would appear that these latter two resins are the most promising for acceptable performance in plastic springs.

## Effect of Glass Reinforcement

The modulus of rigidity of the nonreinforced plastic rods at room temperature generally ranges from 150,000 to over 350,000 pounds per square inch. These values appear low when compared with the 11,000,000 pounds per square inch of spring steel but glass fiber reinforcement can be used to provide marked improvement in torsional properties. The modulus of rigidity of the reinforced springs can be determined by means of the formula (3):

$$\delta = 8 P D^3 n / d^4 G \qquad (2)$$

where $\delta$ = total deflection (in.), P, the load (lb), D the mean coil diameter (in.) (outside diameter minus the wire diameter), n the number of active coils, d the diameter of the coil wire (in.) and G the modulus of rigidity ($lb/in^2$).

Then $$\delta = k_i D^3 n / d^4 \qquad (3)$$

for $$k_i = 8 P / G \qquad (4)$$

One of the more successful glass-reinforced epoxy springs which was produced in quantity gave 0.5-inch deflection at a load of 25 lb. The mean coil diameter of these springs was 0.75-in. and the coil wire diameter 3/16-in. Substituting in equation (3) gives $k_i = 2.93 \times 10^4$. Then from these relationships G is found to be approximately 680,000 $lb/in^2$. The conventional form of the equations used in the computation, however, is based on the assumption that the wire is stressed only in torsion. They do not include the additional stress caused by the shear load. This latter will vary with the ratio of mean coil diameter to wire diameter. Wahl (4) has determined correction factors (K) for varying ratios of D/d. For the plastic spring under consideration K = 1.4 and the real modulus of rigidity therefore is slightly less than **$1.0 \times 10^6$ $lb/in^2$**. This spring was composed of an epoxy resin with five double rovings of glass (a total of 600 ends), cured with 15 percent *m*-phenylenediamine (NBS Composition 23 in Table 1). The resin was brought to a B-stage after 3 hours at 70°C. The tubing was then removed and the springs completely cured by heating at 60°C for 64 hours or longer and 8-10 hours at 150°C. Nonreinforced rods of the cured resin have a modulus of about **250,000 $lb/in^2$**, showing a four-fold increase due to the presence of the glass filaments in the spring. Values for the modulus of rigidity for rods in Table 1 indicate that at least one other epoxy formulation compares favorably with the epoxy resin cured with 15 percent *m*-phenylenediamine (Composition 23). This latter, however is adequate for meeting specification requirements 5 and 6. Typical springs of this type are shown in Figure 1.

Over a hundred of these springs have been made. Under a load **of 25 pounds** they have an average deflection of **0.53-in.** with a standard deviation **of 0.05-in.** Apparently a high degree of homogeneity in static properties is possible in the springs produced by this procedure.

## Properties of Experimental Springs

A number of experimental springs are characterized in Table 3. The springs were compressed to their solid lengths and held in compression for a minimum of 13 days at 135°F. The force required to compress them and the recoverable energy before and after this storage at maximum deflection is given in Table 4. Test results obtained with one epoxy resin, (Composition 23) showed that it was possible to make a helical spring 3-inches long and 1-inch in diameter that would retain over 5-in.-lb of energy (43%) after being held at its solid length for 13 days at 135°F. Since the energy decay curve of a helical spring is an exponential one, the greatest loss of energy occurs during the first few days. A residual energy after thirty days of over thirty percent, therefore, would appear quite possible.

Upon retesting glass-epoxy springs it was noted that the second test invariably resulted in higher recoverable energy values than the first as shown in Table 5. This

**Table 1. Modulus of Rigidity of Plastic Rods**

| NBS Composition Designation | Resin | Modulus of Rigidity -40°F $lb/in^2$ | 73°F $lb/in^2$ | 135°F $lb/in^2$ |
|---|---|---|---|---|
| 1 | Polyester | 437,000 | 156,000 | (a) |
| 2 | Polyester | 348,000 | 164,000 | 106,000 |
| 3 | Polyester | 314,000 | 163,000 | 102,000 |
| 4 | Polyester | 342,000 | 165,000 | 110,000 |
| 5 | Polyester | 406,000 | 235,000 | (a) |
| 6 | Polyester | 376,000 | 249,000 | 99,000 |
| 7 | Polyester | 465,000 | 203,000 | 70,000 |
| 8 | Polyester | 409,000 | 238,000 | 124,000 |
| 9 | Polyester | 435,000 | 252,000 | 162,000 |
| 10 | Polyester | 407,000 | 248,000 | 139,000 |
| 11 | Polyester | 413,000 | 251,000 | 137,000 |
| 12 | Polyester | 498,000 | 248,000 | 115,000 |
| 13 | Polyester | (b) | (a) | (a) |
| 14 | Polyester | 483,000 | 76,000 | (a) |
| 15 | Polyester | 450,000 | 352,000 | 122,000 |
| 16 | Polyester | 500,000 | 274,000 | (a) |
| 17 | Polyester | 457,000 | 334,000 | 195,000 |
| 18 | Polyester | 425,000 | 312,000 | 110,000 |
| 19 | Polyester | 433,000 | 137,000 | (a) |
| 20 | Polyester | 335,000 | 128,000 | (a) |
| 21 | Polyester | 327,000 | 128,000 | (a) |
| 22 | Polyester | 442,000 | 346,000 | 147,000 |
| 23 | Epoxy | 378,000 | 264,000 | 211,000 |
| 24 | Epoxy | 351,000 | 244,000 | 206,000 |
| 25 | Epoxy | 348,000 | 213,000 | 202,000 |
| 26 | Epoxy | 375,000 | 232,000 | 239,000 |
| 27 | Epoxy | 352,000 | 220,000 | 270,000 |
| 28 | Epoxy | 332,000 | 212,000 | 183,000 |
| 29 | Epoxy | 311,000 | 147,000 | 150,000 |
| 30 | Epoxy | (b) | (a) | (a) |
| 31 | Epoxy | (b) | 224,000 | (a) |
| 32 | Epoxy | (b) | 256,000 | (a) |
| 33 | Epoxy | (b) | 248,000 | (a) |
| 34 | Epoxy | (b) | 275,000 | 231,000 |
| 35 | Epoxy | (b) | 170,000 | (a) |
| 36 | Epoxy | (b) | 239,000 | (a) |
| 37 | Epoxy | (b) | 254,000 | 153,000 |
| 38 | Epoxy | (b) | 209,000 | (a) |
| 39 | Epoxy | (b) | 251,000 | (a) |
| 40 | Epoxy | (b) | 237,000 | (a) |
| 41 | Epoxy | (b) | 256,000 | (a) |
| 42 | Epoxy | (b) | (a) | (a) |

*a. Too flexible for test*

*b. Test not performed at this temperature because of flexibility at 135°F.*

**Table 2. Modulus of Rigidity for Plastic Materials**

| Material | Temperature °C | Modulus of Rigidity (a) $lb/in^2$ |
|---|---|---|
| Polystyrene | 30 | 190,000 |
| | 50 | 166,000 |
| | 70 | 137,000 |
| Cellulose acetate | 30 | 63,400 |
| | 50 | 35,400 |
| | 70 | 18,600 |
| Cellulose acetate butyrate | 30 | 63,400 |
| | 50 | 37,300 |
| | 70 | 21,300 |
| Polymethyl methacrylate | 30 | 159,000 |
| | 50 | 117,000 |
| | 70 | 83,000 |
| Wood flour filled phenolic | 30 | 345,000 |

characteristic "work tempering" of the epoxide resins was substantiated by the results of torsion tests on non-reinforced plastic rods. An improvement of up to 120 percent over the original available energy is indicated. Preloading the epoxide spring during storage or within thirty days before use would make it possible to utilize this property of the material.

Glass roving reinforcement increased the potential energy capacity of the springs but it did not increase the percentage of energy recovered after exposure in the compressed state at an elevated temperature. A spring without glass reinforcement retained a certain percentage of energy and this percentage remained essentially constant when its total potential energy was increased due to the introduction of the glass roving. These results lend support to the hypothesis that the creep in the springs is related directly to the actual deformation rather than to the applied force. Attempts were made to introduce the glass roving in such a form that torsion of the rod would produce stress in the glass. This was accomplished by braiding or producing knots in the glass fiber reinforcement used in the springs. Although this procedure caused local variations and irregularities in the density of the spring, the gain in elastic recovery more than compensated for any loss in the maximum strength of the spring. A seven percent increase in recovery appeared to be attributable to the change in the form of the glass roving. There was no comparable improvement in spring properties resulting from the use of a number of glass finishes on the rovings. However, it must be kept in mind that these finishes are, in general, intended to promote the maintenance of strength under somewhat different atmospheric conditions than were experienced in this work.

An analysis of the glass-resin content of some rods and springs is given in Table 6. Both springs and rods showed little variation from end to end and it would appear that springs with a uniform and selected composition can be easily produced. Examination of the cross section of finished springs shows a concentration of glass reinforcing toward the inner periphery of the wires. This can be minimized by first subjecting the filled tubing to an opposing twist when wrapping it around the mandrel

(Please turn to page 50)

## Table 3. Compositions of Some Non-Metallic Springs

| NBS Spring Number | Resin | NBS Composition Designation | Number of Double Rovings[a] | Cure: Initial Time hr | Cure: Initial Temp °C | Cure: Post Time hr | Cure: Post Temp °C |
|---|---|---|---|---|---|---|---|
| 1 | Epoxy | 28 | 6 | 3 | 75 | | |
| 2 | Epoxy | 28 | (b) | 4 | 62 | 48 | 100 |
| 3 | Epoxy | 28 | 6 braided | 3 | 75 | | |
| | | | | 15 | 57 | 2 | 120 |
| 4 | Epoxy | 28 | 10 ribbon tape | 3 | 75 | | |
| | | | | 15 | 57 | 2 | 120 |
| 5 | Epoxy | 28 | 6 | Same as 4 | | | |
| 6 | Epoxy | 28 | 6 | Same as 4 | | | |
| 7 | Epoxy | 28 | 7 | Same as 4 | | | |
| 8 | Epoxy | 28 | 7 | Same as 4 | | | |
| 9 | Epoxy | 23 | 7 | 3 | 75 | 2 | 120 |
| | | | | | | 4 | 150 |
| 10 | Epoxy | 23 | 7 | 3 | 75 | 2 | 120 |
| | | | | | | 4 | 150 |
| 11 | Epoxy | 28 | 9 braided | Same as 4 | | | |
| 12 | Polyester | 43 | 9 braided | 1 | 70 | 1/2 | 120 |
| 13 | Polyester | 44 | 6 | 4 | 23 | | |
| | | | | 2 | 60 | 1 | 84 |
| 14 | Polyester | 44 | 6 | 4 | 23 | | |
| | | | | 2 | 60 | 1 | 84 |
| 15 | Polyester | 44 | 7 | 4 | 23 | | |
| | | | | 2 | 60 | 1 | 84 |

a. A cord of glass roving with 60 ends and a chrome finish doubled and drawn through the tube to make the spring. The wire diameter was ¼-inches.

b. Eighteen double loops of nylon made of 16 ends, 840 denier, 140 filaments, type 300.

## Table 4. Energy Recovery of Some Plastic Springs

| NBS Spring Number | Initial Force | Initial Energy | Final Force | Final Energy[a] | Energy Available |
|---|---|---|---|---|---|
| | lb | in.-lb | lb | in.-lb | % |
| 1 | 28.2 | 11.0 | 14.9 | 3.64 | 33.0 |
| 2 | 21.9 | 6.35 | 13.6 | 1.43 | 22.5 |
| 3 | 36.2 | 9.2 | 23.2 | 4.17 | 45.0 |
| 4 | 36.1 | 5.6 | 25.4 | 2.67 | 48.0 |
| 5 | 40.1 | 12.33 | 11.0 | 0.88 | 7.1 |
| 6 | 42.2 | 15.82 | 14.7 | 1.10 | 7.0 |
| 7 | 43.9 | 12.75 | 24.6 | 1.72 | 13.5 |
| 8 | 43.4 | 11.72 | 14.4 | 1.08 | 9.2 |
| 9 | 43.3 | 15.2 | 23.4 | 3.74 | 24.6 |
| 10 | 40.7 | 12.3 | 37.4 | 5.24 | 42.6 |
| 12 | 6.0 | 1.35 | 4.0 | 0.66 | 50.0 |
| 13 | 42.1 | 15.82 | 12.6 | 1.45 | 9.2 |
| 14 | 37.0 | 9.93 | 18.0 | 1.26 | 12.7 |
| 15 | 36.8 | 14.54 | 14.7 | 1.76 | 12.1 |

a. Inch-pounds available in static load measurements after being compressed to solid length for a minimum of 13 days at 135°F.

## Table 5. Results of Retesting Some Epoxy-Glass Plastic Springs

| NBS Spring Number | Test | Period Between Test 1 and Test 2 | Initial Force | Initial Energy | Final Force | Final Energy[a] | Energy Available |
|---|---|---|---|---|---|---|---|
| | | days | in. | in.-lb | in. | in.-lb | % |
| 1 | 1 | -- | 19.5 | 7.8 | 9.2 | 1.43 | 18.3 |
| 1 | 2 | 45 | 28.2 | 11.0 | 14.9 | 3.64 | 33.0 |
| 3 | 1 | -- | 38.9 | 15.0 | 19.3 | 3.08 | 20.6 |
| 3 | 2 | 14 | 36.2 | 9.2 | 23.2 | 4.17 | 45.0 |
| 4 | 1 | -- | 39.2 | 6.85 | 20.1 | 1.51 | 22.0 |
| 4 | 2 | 7 | 36.1 | 5.6 | 25.4 | 2.67 | 48.0 |
| 11 | 1 | -- | 30.6 | 5.81 | 17.8 | 1.69 | 29.1 |
| 11 | 2 | 45 | 45.1 | 12.2 | 30.2 | 5.4 | 44.5 |

a. Inch-pounds available in static load measurements after being compressed to solid length for a minimum of 13 days at 135°F.

Fig. 1 Typical Collapsing Pressure Failures of Hemispherical Shapes

# Reinforced Plastics in The Construction of External Pressure Vessels

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SEVERAL APPLICATIONS OF reinforced plastics as structural materials have been made in the last decade. They have been applied to car bodies, aircraft, internal pressure vessels, boat hulls, piping, etc. The Navy too has a requirement for a primary plastics structure, one which will perform under severe external hydrostatic loadings. These lightweight housings must also exhibit corrosion resistance, non-magnetic characteristics and high electrical resistivity.

Only limited experience has been had at the Naval Ordnance Laboratory in the use of glass reinforced plastics for constructing external pressure vessels. This experience has provided specific design data but not a broad background of information. To broaden this background, the Laboratory is currently evaluating the characteristics of a variety of laminating resins, glass reinforcements and fabrication techniques suitable for pressure vessel construction. The basic shapes being studied are those of a hemisphere, both with and without a flattened apex, and of a circular cylinder. These two shapes form the basis for assembling models for further testing.

No basic formulas have been developed for calculating the collapsing pressure resistances of vessels constructed of anisotropic materials such as reinforced plastics. Formulas exist for metal structures, and these have been adopted for the plastics work. Roark's formula is used for hemispherical shapes:

$$P_c = 2Et^2/[r^2[3(1-\mu^2)]^{1/2}]$$

where $P_c$ is the collapsing pressure, E is the effective modulus of elasticity, t is the wall thickness, r is the radius and $\mu$ is Poisson's ratio. For short cylinders (L = 6D maximum) under uniform external radial and axial pressures the Trilling-Windenburg formula is used:

$$P_c=[2.42\,E/(1-\mu^2)^{3/2}][t/D^{5/2}/(L/D-0.45\,(t/D)^{1/2})]$$

where t/D is the thickness-to-diameter ratio and L/D is the length-to-diameter ratio. Poisson's ratio ($\mu$) has been determined as equal to 0.3 in previous experimental work on reinforced plastic cylinders (reference (a)) and has been considered as a constant for this work. The Trilling-Windenburg formula gives good results when the failures are of the instability type, where excessive deformations give rise to highly stressed areas. When the failure is by yield of the material without appreciable deformation, the formula is not strictly applicable. Nevertheless, it has been used empirically to permit calculations of experimental vessels of known dimensions. This required the collapsing of cylinders of varying L/D and t/D ratios and the calculation of E to demonstrate its characteristic variations.

Reinforcements, resins and geometry were evaluated in hemispherical form, with results as given in Table I. It is readily seen that a glass fabric gives higher strengths than a glass mat and that polyester and epoxy resins are about equivalent. Variations in geometry, particularly wall thickness, greatly affect the ability of the structure to resist external pressure. On the other hand, a flattened apex can be introduced without affecting strength as long as the wall thickness variations are gradual and the thickness of the flat is sufficient to resist bending. Figure 1 shows some typical failures of hemispheres due to external hydrostatic loading.

## Table 1

### Collapsing Pressure Resistance and Effective Moduli of Reinforced Plastics Hemispherical Structures

| Construction | Wall Thickness Inches | Type | Collapsing Pressure, psi | Effective Modulus, psi x $10^{-6}$ |
|---|---|---|---|---|
| 181-Volan, Polyester | 1/8 | True | 870 | 1.24 |
| " | " | " | 940 | 1.21 |
| " | " | " | 920 | 1.15 |
| " | 1/4 | " | 2300 | 0.77 |
| " | " | " | 2530 | 0.85 |
| " | " | " | 2330 | 0.74 |
| " | " | Flat | 2400 | 0.83 |
| " | " | " | 2400 | 0.81 |
| 181-Volan, Epoxy | " | True | 2270 | 0.76 |
| " | " | " | 2100 | 0.71 |
| 1000-Garan, Polyester | " | " | 1890 | 0.64 |
| " | " | " | 1630 | 0.55 |
| Mech-Mat; Polyester | " | " | 1240 | 0.43 |
| Air Preform Glass, Polyester | " | " | 1720 | 0.58 |
| " | " | " | 1700 | 0.57 |
| Air Preform Glass, Epoxy | " | " | 1780 | 0.60 |
| " | " | " | 1550 | 0.52 |

Inside diameter of hemispheres was 9.86. The "true" hemispheres were of constant thickness and radius while the "flat" units had a 5/8" thick flat section of 4 3/8" diameter at the apex.

When the data of Table I are compared with the data of Figure 8, it is seen that a hemisphere can be made considerably stronger than a ribbed cylindrical sidewall. Therefore, the strength of the sidewall is of major importance in the design of any external pressure vessel fabricated from these two basic shapes. As a result, studies have concentrated on the cylindrical section so as to determine the optimum design parameters and selections of materials.

The initial series of experiments was with small diameter convolutely wound cylindrical pressure vessels, as shown in Figure 2. Figure 3 shows data for style 181 glass fabric reinforced polyester tubes. It is readily seen that $P_c$ increases with a decrease in the L/D ratio and with an increase in the t/D ratio. Here then are two ways by which the strength of the structure may be controlled. It should also be noted that the slopes of these curves change markedly in the range of L/D ratios from 1 to 2. At L/D ratios above this range, failure in the specimen is of a typical instability nature, while below the failure is typically yield. Probably both the yield and the instability mechanisms exert their influence in the intermediate range. At extremely low L/D ratios the axial compression strength of the cylinder appears to be the yield mechanism of importance.

Once $P_c$ is determined on a series of tubes of known t/D and L/D ratios, E may be calculated. A chart of E versus L/D for various t/D's, such as in Figure 4, allows the estimation of $P_c$ for experimental cylinders. The drop in E at low L/D ratios again illustrates that the formula is not applicable to failures of a yield nature. It can, however, be used empirically by introducing an estimated E consistent with the specific t/D and L/D ratio of the pro-

Fig. 2 Typical Collapsing Pressure Failures of Cylindrical Shapes.

Fig. 3

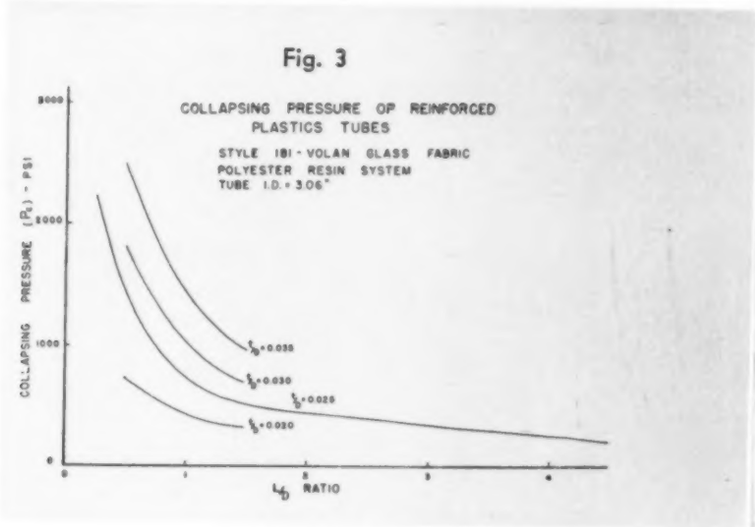



Fig. 4

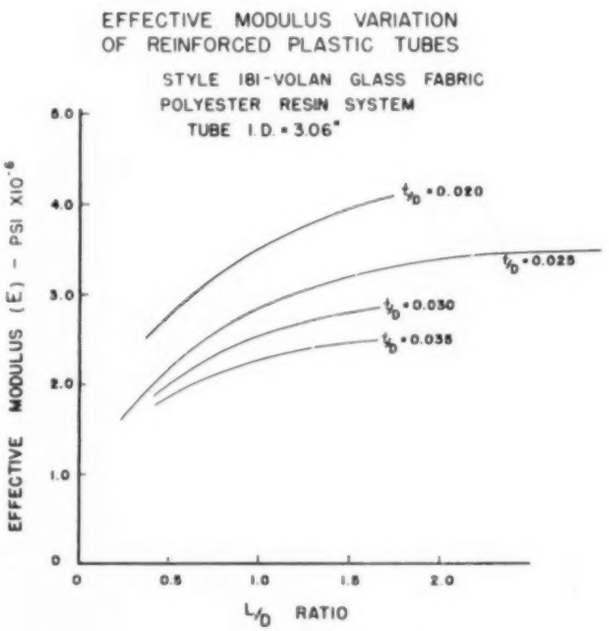


Fig. 5

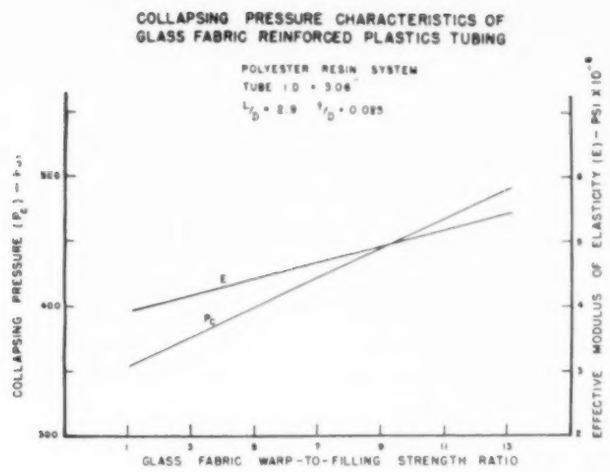
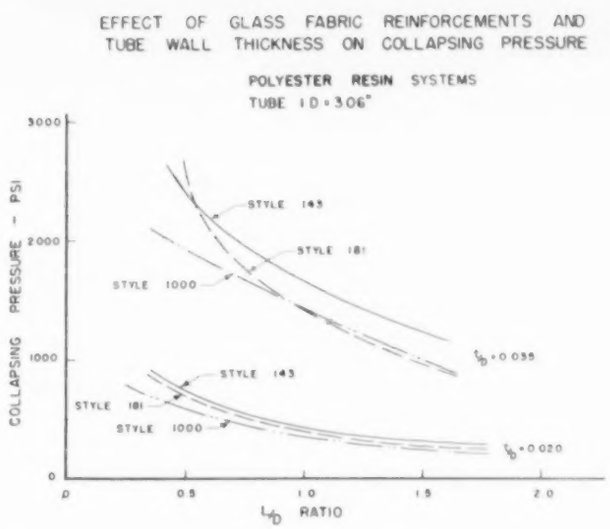

COLLAPSING PRESSURE CHARACTERISTICS OF GLASS FABRIC REINFORCED PLASTICS TUBING
POLYESTER RESIN SYSTEM
TUBE I.D. = 3.06"
L/D = 2.9 t/D = 0.035
COLLAPSING PRESSURE (Pc) - PSI
EFFECTIVE MODULUS OF ELASTICITY (E) - PSI X 10⁻⁶
E
Pc
GLASS FABRIC WARP-TO-FILLING STRENGTH RATIO

Fig. 6




posed vessel.

It is reasonably expected that changes in the properties of the glass reinforcing fabric would cause proportional changes in the pressure resistance capabilities of a plastic structure. Figure 5 is a plot of $P_c$ and E versus the fabric warp-to-filling strength ratio. It is shown that the properties of the fabric directly relate to the properties of the structure under external pressure loading. Although style 143 fabric, which imparts a high hoop strength to the tube, gave the highest $P_c$ and E results, there is still another factor to be considered before a final choice is made. Axial strength may be of great importance depending upon the use of scarf or sleave adhesive joints in the fabrication process and upon the handling given the unit, or because of structural axial strength requirements other than to resist external pressure. Test data show that the axial compression strength of polyester tubes reinforced with 181 fabric is 30,000 psi, while with 143 fabric it is 16,000 psi. If an epoxy resin is substituted, the 143 fabric will give a compression strength of from 18,500 to 21,500 psi. Therefore, a fabric other than 143 might be a better selection, and this possibility should be carefully examined for each application.

Additional data on the effectiveness of glass fabric as a reinforcement are given by Figure 6. It is to be noted that the greatest differences between reinforcements exist at the maximum wall thickness where the effective modulus is lowest. Table II shows that the thickness of the reinforcing fabric also plays a small role in determining the strength of a cylinder. The greatest variations occur in the heavier wall thickness and at the highest L/D ratios where the heavier fabrics seem to give slightly stronger tubes. Other strength differences can be expected if the resin is varied. Table III shows that a heat-resistant polyester resin gave the highest results, while an epoxy was medium and a general-purpose polyester was lowest. It was expected that glass finish and curing agent would also affect collapsing pressure resistance. This did not prove to be true, as the data of Table

## TABLE II

### Variation of Collapsing Pressure Resistance And Effective Modulus With Glass Fabric

| Tube Geometry | | Materials | | | | | |
|---|---|---|---|---|---|---|---|
| L/D | t/D | 181-Volan | | 182-Volan | | 184-Volan | |
| | | $P_c$ psi | E psi x $10^{-6}$ | $P_c$ psi | E psi x $10^{-6}$ | $P_c$ psi | E psi x $10^{-6}$ |
| 1.5 | 0.025 | 500 | 3.20 | 600 | 3.35 | 550 | 3.20 |
| | 0.035 | 1000 | 2.45 | 1350 | 3.15 | 1250 | 2.95 |
| 1.0 | 0.025 | 750 | 2.85 | 850 | 3.05 | 800 | 2.90 |
| | 0.035 | 1450 | 2.30 | 1800 | 2.75 | 1850 | 2.65 |
| 0.5 | 0.025 | 1400 | 2.20 | 1550 | 2.65 | 1400 | 2.35 |
| | 0.035 | 2500 | 1.80 | 2900 | 2.05 | 2500 | 1.85 |
| 0.25 | 0.025 | 2200 | 1.60 | 2000 | 1.40 | 1450 | 1.15 |
| | 0.035 | 3100 | 0.85 | 2600 | 0.75 | 2550 | 0.80 |

Tests based on convolutely wound tubes with an I.D. of 3.06. The tubes were laminated with a general purpose polyester resin mixture containing 60% rigid resin, 35% flexible resin and 5% styrene.

## TABLE III

### Variation In Collapsing Pressure Resistance And Effective Modulus With Laminating Resin

| Tube Geometry | | Materials | | | | | |
|---|---|---|---|---|---|---|---|
| L/D | t/D | General Purpose Polyester | | Heat Resistant Polyester | | Epoxy + Fi | |
| | | $P_c$ psi | E psi x $10^{-6}$ | $P_c$ psi | E psi x $10^{-6}$ | $P_c$ psi | E psi x $10^{-6}$ |
| 1.5 | 0.025 | 500 | 3.20 | 700 | 3.25 | 600 | 2.85 |
| | 0.035 | 1000 | 2.45 | 1350 | 3.00 | 1200 | 2.70 |
| 1.0 | 0.025 | 750 | 2.85 | 850 | 3.15 | 800 | 2.60 |
| | 0.035 | 1450 | 2.30 | 1950 | 2.85 | 1750 | 2.45 |
| 0.5 | 0.025 | 1400 | 2.20 | 1700 | 2.60 | 1600 | 2.25 |
| | 0.035 | 2500 | 1.80 | 2900 | 2.00 | 2600 | 1.95 |
| 0.25 | 0.025 | 2200 | 1.60 | 2350 | 1.60 | 2500 | 1.50 |
| | 0.035 | 3100 | 0.85 | 3500 | 0.90 | 3400 | 0.85 |

Tests based on convolutely wound tubes reinforced with 181-Volan glass fabric.
I.D. of tubes = 3.06
The general purpose polyester was formulated from 60% rigid resin, 35% flexible resin and 5% styrene.

## TABLE IV
### Variation In Collapsing Pressure Resistance With Glass Finish and Curing Agent

| Tube Geometry | | | | |
|---|---|---|---|---|
| L/D | t/D | 181-Volan Epoxy + CL | 181-NOL 24 Epoxy + CL | 181-NOL 24 Epoxy + A |
| | | $P_c$ - psi | $P_c$ - psi | $P_c$ - psi |
| 1.5 | 0.025 | 500 | 500 | 550 |
| | 0.035 | 1100 | 1100 | 1100 |
| 1.0 | 0.025 | 750 | 750 | 600 |
| | 0.035 | 1650 | 1750 | 1750 |
| 0.5 | 0.025 | 1350 | 1100 | 1000 |
| | 0.035 | 2750 | 2500 | 2800 |
| 0.25 | 0.025 | 2500 | 2250 | 1900 |
| | 0.035 | 3500 | 3200 | 3500 |

Tests based on convolutely wound tubes with an I.D. of 3.06.

## TABLE V
### Variation In Collapsing Pressure Resistance And Effective Moduli For Glass Roving Reinforced Tubes

| Tube Geometry | | 60° Helix Tubes | | 90° Helix Tubes | |
|---|---|---|---|---|---|
| L/D | t/D | $P_c$ | E | $P_c$ | E |
| | | psi | psi x $10^{-6}$ | psi | psi x $10^{-6}$ |
| 2.9 | 0.025 | 575 | 6.4 | 750 | 7.9 |
| | 0.035 | - | - | - | - |
| 1.5 | 0.025 | 950 | 5.2 | 1100 | 6.3 |
| | 0.035 | 2250 | 5.5 | 2400 | 5.7 |
| 1.0 | 0.025 | 1350 | 4.9 | 1650 | 6.1 |
| | 0.035 | 3300 | 5.1 | 2700 | 4.2 |
| 0.5 | 0.025 | 2800 | 4.4 | 2050 | 3.3 |
| | 0.035 | 4000 | 2.8 | 2800 | 2.0 |
| 0.25 | 0.025 | 2800 | 1.9 | 2150 | 1.4 |
| | 0.035 | 4200 | 1.1 | 2750 | 0.7 |

Tubes were helically wound with 150 - 1/0 114 treated glass rovings and were bonded with an epoxy resin using curing agent A. The inside diameter was 3.06.

## TABLE VI
### Variation of Collapsing Pressure Resistance and Effective Modulus With Increasing Tube Diameter

| Tube I.D. Inches | Collapsing Pressure psi | Effective Modulus psi x $10^{-6}$ |
|---|---|---|
| 3.06 | 355 | 3.90 |
| 6.02 | 350 | 3.85 |
| 9.86 | 350 | 3.81 |

Each value is an average of three tests

L/D = 2.90 t/D = 0.025

Tubes reinforced with 181-Volan glass fabric and bonded with a general purpose polyester resin mixture of 80% rigid resin, 15% flexible resin and 5% styrene.

IV show. It might also be reasoned that soaking of the reinforced plastics tubes in water would materially affect their strength. At first there was some decrease, in the order of 15 or 20%, but after one year they recovered the loss. Even tubes laminated with resins known to be very poor on temperature-humidity cycling showed the strength recovery trend.

The tubes used in the above tests had certain variations. With style 181 fabrics, glass content ranged from 55 to 63%, while with 143 fabric it ranged from 60 to 70%. Tubes bonded with polyester resin, upon collapsing, had coefficients of variation ranging from 1.0% to 15.4%, with an average of 3.8%. When epoxy resins were used, the coefficients ranged from 0.0% to 13.5%, with an average of 4.2%.

Glass rovings may be readily used in a helical pattern to reinforce a cylindrical structure. They give excellent results, as indicated by the variations of $P_c$ and E versus the helix angle of wind pictured in Figure 7. Similarly, as with fabric reinforced tubes, it is noted that as the hoop strength of the tube is increased, pressure resistance and stiffness increase. In contrast to the helical distribution of glass in a piece, the rovings may also be placed so that they lay only in the axial and hoop directions. The technique for doing this is the so called 0°-90° wind. With this technique the axial-hoop strength ratio can be as readily controlled as with helical winding and so vary $P_c$ and E. This effect is shown by the points labeled 2:1 and 4:1 (representing the hoop-to-axial strength ratio of the tube) over the 90° helix line of Figure 7. Certain theoretical advantages accrue by using the 0°-90° wind technique, as the glass is put more nearly in direct line with the applied stresses than when wound helically. It is, therefore, to be expected that these structures would have better stress resisting characteristics under both short-time and long-time loading conditions. Also, at the higher collapsing pressures, which may be required, the maximum axial strengths for the application can be more readily maintained. 0°-90° wound tubes have axial compression strengths of 23,000 psi for a 2:1, axial-to-hoop strength ratio, as compared to 18,000 psi for a 60° helix wind and 15,000 psi for an 80° helix wind. Table V shows the strength variations of roving reinforced tubes with L/D and t/D ratios. The curves plotted from this data are very similar to those of Figures 3 and 4. Glass con-

Fig. 7

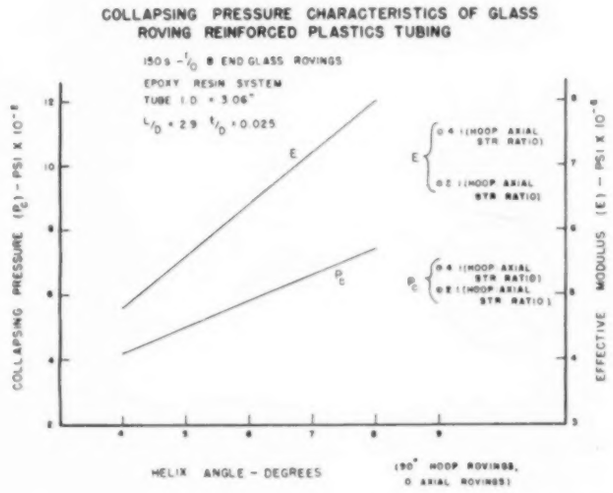
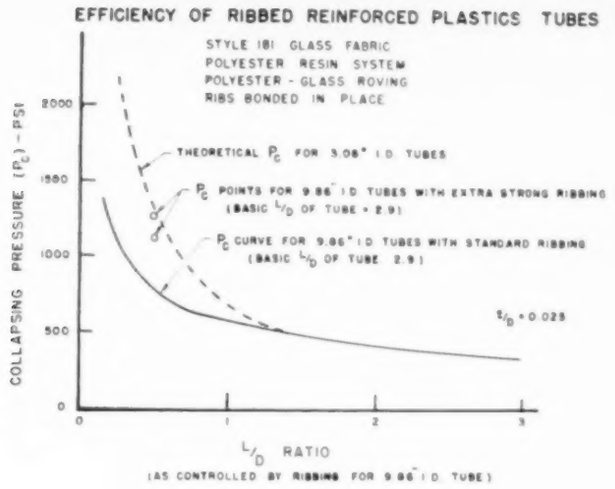

COLLAPSING PRESSURE CHARACTERISTICS OF GLASS ROVING REINFORCED PLASTICS TUBING
150 s - 1/0 B END GLASS ROVINGS
EPOXY RESIN SYSTEM
TUBE I.D. = 3.06"
L/D = 2.9 t/D = 0.025
E
Pc
COLLAPSING PRESSURE (Pc) - PSI X 10⁻⁶
EFFECTIVE MODULUS (E) - PSI X 10⁻⁶
HELIX ANGLE - DEGREES
(90° HOOP ROVINGS, 0 AXIAL ROVINGS)

Figure 8


EFFICIENCY OF RIBBED REINFORCED PLASTICS TUBES
STYLE 181 GLASS FABRIC
POLYESTER RESIN SYSTEM
POLYESTER - GLASS ROVING
RIBS BONDED IN PLACE
THEORETICAL $P_c$ FOR 9.86" I.D. TUBES
$P_c$ POINTS FOR 9.86" I.D. TUBES WITH EXTRA STRONG RIBBING (BASIC $L/D$ OF TUBE = 2.9)
$P_c$ CURVE FOR 9.86" I.D. TUBES WITH STANDARD RIBBING (BASIC $L/D$ OF TUBE 2.9)
$t/D$ = 0.025
COLLAPSING PRESSURE ($P_c$) - PSI
2000
1500
1000
500
0
0
1
2
3
$L/D$ RATIO
(AS CONTROLLED BY RIBBING FOR 9.86" I.D. TUBE)

tents for the roving reinforced plastics tubes ranged from 82 to 84%. The coefficients of variation of the test data ranged from 0.3% to 5.2%, with an average of 2.5%.

Very limited work has been done on bag-molded glass mat reinforced tubes. Effective modulii of 3.5 x $10^6$ psi, 2.2 x $10^6$ psi, 2.0 x $10^6$ psi and 1.9 x $10^6$ psi at L/D ratios respectively of 1.5, 1.0, 0.5 and 0.25 have been measured on tubes having a t/D of 0.0125. These data indicate that the glass mat construction should certainly be considered for external pressure resistance applications.

Scale factor is an important question in the use of all the above data. A series of data, Table VI, show that the scale factor is effectively 1:1, as would be predicted from the Trilling-Windenburg formula. Since any increase in the radii of a tube approaches the ideal laminating conditions of a flat sheet, it is expected that the 1:1 scale factor ratio will hold true at diameters larger than those tested to date.

In the practical application, a cylinder will generally have a specified length, yet its collapsing pressure requirement may demand a much lower effective L/D ratio than that of the basic tube. Because of this, a means of controlling the effective L/D ratio of a structure is needed. Ribs are one possibility, as they will reinforce the tube and establish the effective length by their separation distances. Figure 8 shows that $P_c$ can be controlled in the instability failure region by this method. In the yield region, however, these same ribs, which were designed to fail simultaneously with the sidewall, do not give sufficient support to allow the $P_c$ of the tube to follow the theoretical $P_c$-L/D curve. The units, therefore, failed in instability, as shown in Figure 9. Although the theoretical curve is not followed, the collapsing pressure resistance of the cylinder is increased three-fold without a major weight increase. Figure 8 also shows that further pressure resistance may be developed by increasing the rib cross-section or the number of ribs but only by increasing the weight as well. Increasing the rib size leads to a yield failure, as pictured in Figure 9. In practice, the ribs can be molded integral with the sidewall on a collapsible mandrel rather than adhering them in place, as was done in the Laboratory, and some further strength gains should be had.

Not only must a reinforced plastics pressure vessel have a high resistance to short-term external pressure, but it must be able to resist a high proportion of this

Figure 9

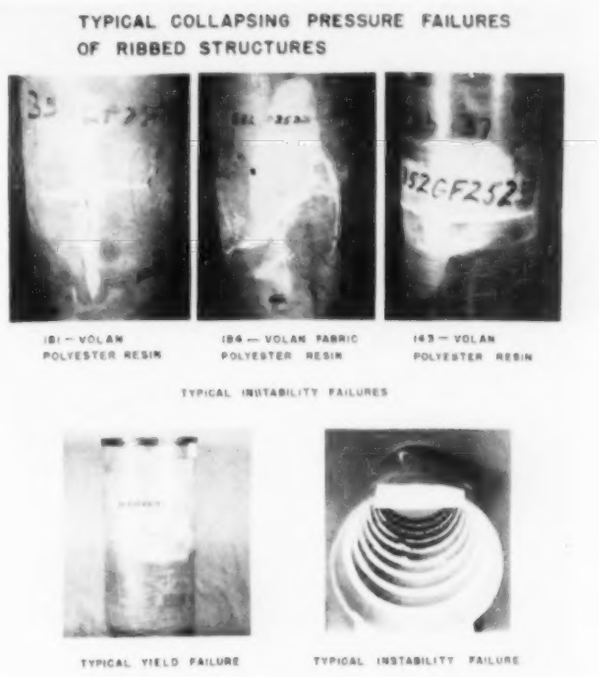
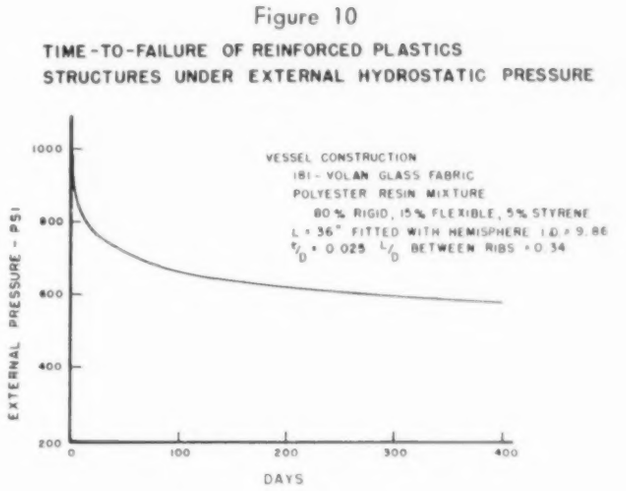

TYPICAL COLLAPSING PRESSURE FAILURES OF RIBBED STRUCTURES
181 — VOLAN POLYESTER RESIN
184 — VOLAN FABRIC POLYESTER RESIN
143 — VOLAN POLYESTER RESIN
TYPICAL INSTABILITY FAILURES
TYPICAL YIELD FAILURE
TYPICAL INSTABILITY FAILURE

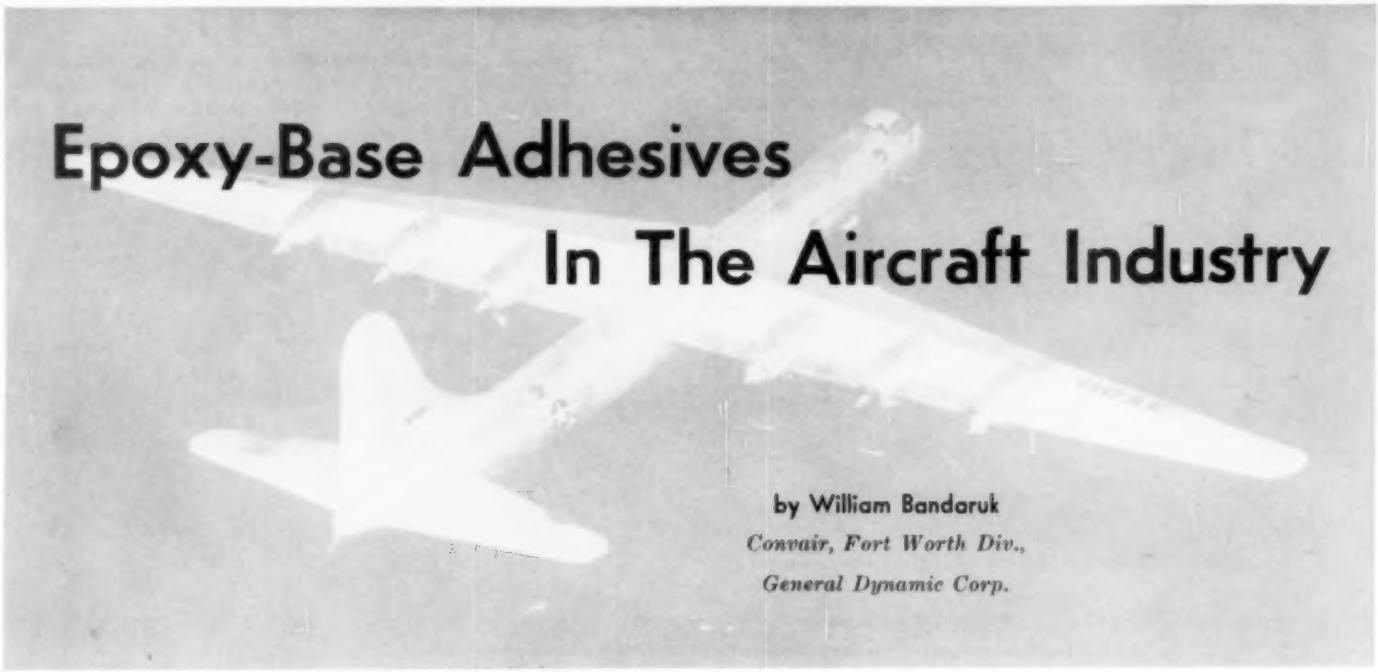
Figure 10




pressure for an extended period of time. Measurements of this characteristic have been made and the data are pictured in Figure 10. Vessels loaded to 80% of their short-term strengths do not withstand continuous pressure for any great length of time. At 60% loading they will last about 300 days. It is indicated that if the load is not over 50% of the short-term strength, the unit will last well in excess of one year. Vessels loaded at 40% of their ultimate strength have been found to maintain their full short-term strength even after one year's long-term loading.

Since the Navy's reinforced plastics housings are to contain instrumentation, they must be capable of maintaining a dry atmosphere on the inside, or at least limit the moisture permeability to a degree which can readily be absorbed by a desiccant. During the long-term pressure tests, the moisture permeability rate of these structures was measured. At 400 psi external pressure the average

(Please turn to page 38)



# Epoxy-Base Adhesives In The Aircraft Industry

by William Bandaruk

*Convair, Fort Worth Div.,*

*General Dynamic Corp.*

**Convair, over the past decade,** has pioneered the use of organic metal-to-metal adhesives as a replacement for conventional fasteners in airframe production. The giant B-36 bomber, the Sea Dart, the Tradewind, the F-102 and the TF-102 are planes in which this type of adhesive has been utilized successfully.

The inherent advantages of organic metal-to-metal adhesives over metal fasteners are these:

1. Production of aerodynamically smooth surfaces which lowers air resistance, an important factor in all planes and a must in making of supersonic planes.
2. Realization of uniform stress over entire bonded surfaces, eliminating local stress concentrations that occur in use of metal fasteners.
3. Utilization of thinner skins as a result of uniform stress, thus resulting in airframe weight reduction. Also, the adhesive is lighter in weight than metal fasteners that it replaces.
4. Fabrication of complex designs have been realized which might not have been possible by mechanical means alone.
5. Increased resistance to fatigue as compared to metal fasteners.
6. Dampening effects causing decrease in vibration.
7. Liquid tightness (water and fuel) can be attained with some adhesives.
8. Finally, reduction of corrosion which is quite frequent when using metal fasteners. Metal-to-metal adhesives must pass severe corrosion tests.

## Largest Bomber

One of the largest uses in the aircraft industry of a metal-to-metal adhesive has been in the production of the B-36. This well known, world's-largest bomber has over one fourth of the exterior area of the wing bonded to stiffeners by means of an organic adhesive (1). Stiffeners are any members supplying stiffness to a sheet (skin). This adhesive consists of neoprene-phenolic-nylon, and is sold under the tradename of Metlbond. The record of success of this adhesive in the B-36 amply demonstrates the advantages of this type fastening for aircraft production.

The success of Metlbond in this sky giant spurred Convair to utilize this type fastening in other aircraft such as the Sea Dart, the Tradewind, the F-102 and the TF-102.

But, with ever increasing speeds demanded of aircraft now in design or production, the metal adhesives used successfully in production of subsonic aircraft were found to be inadequate for supersonic airplanes. The metal adhesives were found to have poor physical properties at the elevated temperatures generated by air friction on the high-speed planes.

Contemplating the use of metal adhesives in design of a supersonic plane, we evaluated a number of high-temperature resistant metal-to-metal adhesives.

An epoxy-phenolic base adhesive was found to be satisfactory for high-temperature applications. This adhesive, known as 422-J, was developed by Shell Development Company under Air Force Contract. The adhesive is manufactured in the form of a 36-inch wide tape, and consists of epon-phenolic resins, metallic filler, and glass cloth carrier. The tape is easy to apply and handles smoothly. It can be obtained as a thin tape (approximately 10 mils) or as a thick tape (20 mils). A disadvantage connected with this adhesive is its short life when stored at room temperature. But, when stored below 40°F, the shelf-life is extended to six months.

We have evaluated (2) this adhesive in accordance with Air Force Specification MIL-A-8331. This is the specification to which metal-to-metal adhesives must qualify for use in U.S. Air Force planes. Many of you may be familiar with its stringent requirements. In addition to evaluating 422-J to this specification, shear strengths were obtained at temperatures up to 500°F. The results of this evaluation are compared to the requirements shown in Table I.

Examination of Table I shows that this epoxy-phenolic adhesive does not have as high shear strength at room temperature as some other commercial adhesives. Some commercial adhesives display room temperature shear

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stress of approximately 3500 psi, but their shear stress decreases rapidly with temperature increase. However, the shear stress (shear strength per square inch) of 422-J tape falls off gradually from — 68 F to 500 F, rather than abruptly. This property is particularly appreciated by aircraft design and structures engineers. In addition, this adhesive had properties that can be reproduced more consistently than metal adhesives containing rubber as the plasticizer for the base resin.

Because of its volatile content, stemming from solvents and reaction products given off during cure, this adhesive was found not to form liquid tight bonds such as are desired in particular applications.

## Designer's Dream

This epoxy-phenolic base adhesive has made another designers' dream possible. The design referred to is a sandwich panel of high structure characteristics, consisting of a metal skin bonded on each side of special core. In the aircraft industry we refer to any thin sheet of metal as skin. The realization is a sandwich panel that has higher stiffness-to-weight ratio than those of conventional construction. Furthermore, these sandwich panels have been subjected to rugged static tests and fatigue loads at high temperatures and the results have been good.

The aircraft industry has found many other uses for epoxy-base adhesives. Fiberglas laminates have been bonded to fiberglas laminates and to aluminum with an epoxy adhesive which has an initial paste-like consistency. In the bonding of strain gages in various structure test programs, very thin coats of a formulation containing epoxy resins has been found satisfactory.

In the bonding of special type stator rings for electrical motors, Epon VI replaced an expensive thin adhesive film, resulting in an approximate $15,000 first-year tangible savings in production. This was over and above the cost of special jigging required to hold these rings during bonding. This does not include the considerable intangible goodwill from the Air Force.

Epoxy-base adhesives have also been found highly versatile in many minor applications where curing anywhere from room temperature to high temperature is desired.

### TABLE I

**Evaluation Tests of Shell's 422J Tape and Requirements of Specification MIL-A-8331**

| Test | Condition | Spec. MIL-A-8331 Minimum Requirements | Convair's Average Results | Number of Specimens |
|---|---|---|---|---|
| 1: Shear Strength | Room Temp. | 2500 psi | 2749 psi | 30 |
| 2: Shear Strength | 180°F | 1250 psi | 2441 psi | 15 |
| 3: Shear Strength | 260°F | Not part of Spec. | 2285 psi | 15 |
| 4: Shear Strength | 350°F | Not part of Spec. | 1944 psi | 15 |
| 5: Shear Strength | 500°F | Not part of Spec. | 1733 psi | 15 |
| 6: Shear Strength | -68°F | 2500 psi | 3177 psi | 15 |
| 7: Vibration Fatigue (10 Million Cycles) | Room Temp. | 650 psi | 650 psi | 3 |
| 8: Creep Rupture | Room Temp. 200 Hours | 1600 psi | 1600 psi | 6 |
| 9: Creep Rupture | 180°F, 200 Hours | 800 psi | 800 psi | 6 |
| 10: Bend Test | Room Temp. | 150 lbs | 151 lbs | 15 |
| 11: Storage Test (6 months 35°F or lower) Shear Strength | Room Temp. 260°F | | 2475 psi 2050 psi | |
| Fluid Soaks | | | | |
| 12: Shear Strength | Salt Spray 30 days | 2000 psi | 2504 psi | 15 |
| 13: Shear Strength | Tap Water 30 days | 2000 psi | 2515 psi | 10 |
| 14: Shear Strength | Ethylene Glycol 7 days | 2000 psi | 2556 psi | 10 |
| 15: Shear Strength | Anti-Icing Fluid, 7 days | 2000 psi | 2634 psi | 10 |
| 16: Shear Strength | Hydraulic Oil, 7 days | 2000 psi | 2671 psi | 10 |
| 17: Shear Strength | MIL-H-3136, Type III (Hydrocarbon Fluid 7 days) | 2000 psi | 2543 psi | 10 |
| 18: Shear Strength | JP-4 Fluid, 7 days | 2000 psi | 2510 psi | 10 |

### TABLE II

**Tests On A Liquid Epoxy Resin Cured With Meta Phenylene Diamine Accelerator and Requirements Of Specification MIL-A-8331**

| Test | Condition | Spec. MIL-A-8331 | Convair's Average Results | Number of Specimens |
|---|---|---|---|---|
| 1: Shear Strength | Room Temp. | 2500 | 3023 | 40 |
| 2: Shear Strength | 180°F | 1250 | 3209 | 12 |
| 3: Shear Strength | 260°F | Not part of Spec. | 3353 | 12 |
| 4: Shear Strength | 350°F | Not part of Spec. | 858 | 12 |
| 5: Shear Strength | 500°F | Not part of Spec. | 380 | 12 |
| 6: Shear Strength | -68°F | 2500 | 2949 | 12 |
| 7: Vibration Fatigue (10 million cycles) | Room Temp. | 650 psi | 650 | 9 |
| 8: Creep Rupture | 260°F, 200 Hours | 1200 psi (Not part of Spec.) | 1200 psi | 3 |
| 9: Bend Test | Room Temp. | 150 lbs. | 163 | 8 |
| Fluid Soaks | | | | |
| 10: Shear Strength | Salt Spray, 30 days | 2000 psi | 2955 | 12 |
| 11: Shear Strength | Tap Water, 30 days | 2000 psi | 3422 | 12 |
| 12: Shear Strength | Ethylene Glycol, 7 days | 2000 psi | 3056 | 12 |
| 13: Shear Strength | Anti-Icing Fluid, 7 days | 2000 psi | 3087 | 12 |
| 14: Shear Strength | Hydraulic Oil, 7 days | 2000 psi | 3304 | 12 |
| 15: Shear Strength | MIL-H-3136, Type III, 7 days | 2000 psi | 3010 | 12 |
| 16: Shear Strength | JP-4 Fluid, 7 days | 2000 psi | 3152 | 12 |

In some cases, no pressure can be used; in others, high pressure is needed to press-fit the component parts. The available commercial adhesives and resins can be modified using various curing agents and fillers to fit the conditions of the problem at hand.

The evaluation and qualification of a new adhesive is a major undertaking and quite expensive. In evaluating high-temperature adhesives, we submit the adhesive to rigorous screening tests. The screening tests are the most stringent tests of a complete evaluation.

After an adhesive has passed a full evaluation, the next step is to use it in the fabrication of test parts. The fabricated parts are subjected to simulated environmental conditions of high and low temperatures, vibration, high loads, and whatever tests the design or structures engineer desires the specimen to undergo. Rest assured, these are numerous.

Then, after the evaluation and the environmental tests, the adhesive must be proved to be compatible with production processes and schedules.

After we accept an adhesive for use in the shop, rigorous controls are set up in the laboratory to control the quality of the adhesive. Production must set up high standard quality controls in the bonding of parts in order to be sure that the bonded part meets the design allowables for which it was fabricated.

## The Volatiles Problem

One of the problems in the use of adhesives containing phenol-formaldehyde resins is the formation of volatiles on curing. At the cure temperature of 350°F, the volatiles develop pressures for which provisions must be made to release them, or sufficient press pressure must be applied to contain them within the assembly being bonded. Either provision means that the tooling engineers have gross problems.

One of the major tooling problems is the design of large high-pressure presses for containing these volatiles within an assembly being bonded. Another major problem is making of expensive high-order-tolerance dies for bonding of parts and maintaining this precision throughout the production of a large number of these parts.

Because of the many inherent problems present when using metal-to-metal adhesives containing volatiles (solvents or reaction products), we have a corporation-funded project for developing a high-temperature (500°F) metal-to-metal, and metal-to-core adhesive with the objective being the development of a one hundred percent solids material. With such an adhesive, the following advantages would be realized:

1. Low pressures could be utilized in bonding; thus, bonding could be accomplished using vacuum pressure techniques.

2. Solid fillets would be formed in the bonding of skins to core. This feature is certainly appreciated by structural engineers who do not desire porous fillets in this type of structure. The term fillets is applied to the adhesive cured in the right angle contact between the core and skin.

3. Bonding of large metal-to-metal areas could be realized at moderate pressures.

4. Fuel tight bonds would be a certainty.

Moreover, it would be desirable if this target adhesive could be cured at low temperatures (300°F); thus, rubber blankets that are used during bonding could be used over and over many times more than when bonding at 350°F.

It would be an advantage if this 100% solids adhesive could be in the form of a tape. If processing is not too difficult and is reproducible, certainly paste or liquid would be satisfactory.

## Reasons for Epoxy

In reviewing the many base resins which could possibly be used in this development program, the epoxy resin was chosen for these reasons:

1. Adhesion characteristics displayed by hydroxyl groups formed from the epoxy group upon curing with amines. In addition, there are hydroxyl groups in the molecule of this class of resin which impart adhesion.

2. No volatiles (solvents or reaction products) are evolved when this resin is cured with amines. This property would prevent formation of voids in the bond, and low-pressure bonding could be used satisfactorily.

3. Curing with amines produces low shrinkage which should minimize the stresses in the bond.

4. This resin possesses inherent wetting characteristics, which is very desirable during cure of parts. The adhesive should flow to fill all the voids. Good wetting adds to molecular attraction between the adhesive and adherends. In addition, wetting aids in decreasing stress concentrations in the bond.

5. When cured with an amine and heat, the polymer formed has an inherent toughness.

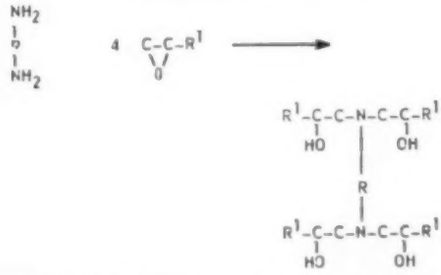
6. Past experience has shown that good reproducibility and repeatability can be attained with this resin.

A liquid epoxy resin was selected for this program since it readily lends itself to formulation with accelerators, fillers, other resins, and the many chemicals required in this kind of development.

An aryl diamine was found to be the amine with which to cure the epoxy resin to obtain more promising high temperature results than other classes of amines. The aromatic portion of the diamine should aid in the attaining of high-temperature resistance.

The polyfunctional amines cure the epoxy compounds containing one epoxy group by the following general reaction:

**General Reaction of Epoxy Compounds with Poly Functional Amines**



*Where the R1 contains epoxy groups, such as in epoxy resins containing an epoxy at both ends, the reaction further continues to form cross linked thermoset polymers.*

We have been encouraged in this endeavor by the evaluation of an adhesive made from a liquid epoxy resin and metal phenylene diamine accelerator, with a fiberglass cloth carrier for reinforcement and glueline spacer.

Samples of this formulation were heated in open dishes at 350°F for several hours and found to contain only 0.3% volatiles. This low volatile content certainly is sufficiently close to the target of 100% solids.

This material was tested according to Specification MIL-A-8331 (3). The values obtained are compiled in Table II.

Other findings of interest with this adhesive system were; no creep at 260°F for 200 hours at 1600 psi, and no creep at 260°F for 100 hours at loads of 2000 psi to 2200 psi. Creep is a measurement of the glueline flow when subjected to prolonged static load.

In aluminum skin-to-core application, the adhesive made solid fillets, and displayed good adhesion as evidenced by punch-out tests at 260°F. This material was also found to form liquid-tight bonds.

To ascertain the adhesive's ability to bond large overlaps, two 19" x 20" x 0.040" clad aluminum skins were bonded congruent with each other at 300°F, 100 psi, 45 minutes. This aluminum laminated panel was cut into 51 specimens (1" x 6") which were made into shear strength speciments as follows: staggered cuts were made at the approximate center of the one-inch wide specimens. One cut was made through one skin and the glueline, just to the other skin. The second cut was made similarly on the other side of the specimen so as to form ½" x 1" bonded area for shear strength test. These specimens were tested for shear stress at -70°F, room temperature, and 260°F, and at a loading rate of 600 lbs./minute. Results from these tests are shown in Table III.

In experimenting with this liquid epoxy adhesive, we found that "B" staging the mixture, after placement in the bond, caused considerable improvement of shear stress at room temperature and 260°F. The values substantiating this conclusion are shown in Table IV.

## Age Before Cure

The values in Table V establish that the adhesive should be allowed to "B" stage for 16 to 24 hours before curing. With this in mind, a number of shear stress specimens were made using standard clad aluminum specimens for the purpose of determining the reproducibility of results and ascertaining the temperature at which the values fall off. All standard panels were made using the adhesive as soon as it was made. All panels were bonded within 16 to 24 hours after lay-up. The test findings are given in Table V.

At first we did not realize the extent to which it is important to maintain the concentration of the meta phenylene diamine curing agent until the concentration was varied from 12 phr (parts per hundred resin) to 17 phr. Standard ½-inch lap shear panels were prepared and tested in accordance with Specification MIL-A-8331.

The results of this series of tests are compiled in Table VI. The optimum concentration of this accelerator is 15 phr.

One prime disadvantage with cured epoxy adhesives is the low inherent peel strength. Incorporation of polysulfides or polyamides does increase the peel characteristics, but this is accompanied by appreciable lowering of shear strength at high temperature.

Whether or not a long shelf-life tape containing the target properties of a 100% solids adhesive can be realized is a challenge to the epoxy chemists.

It is difficult to realize but before 1941 the epoxy resin was not known. A systematic search was organized by Devoe and Reynolds for a resin that was only an idea created in 1939 (4). But by 1941 this resin had been synthesized. Primarily, this company was interested in this resin for protective coating bases.

In summary, the epoxy-base adhesives have made great strides in aircraft applications since they were first introduced into this industry not too many years ago. The

(Please turn to page 50)

### TABLE III
**Shear Stress At Various Temperatures of Specimens Cut From Bonded Skins With Large Overlap**

| Temp. of Test* | Shear Strength Average | Max. | Min. | No. of Specimens |
|---|---|---|---|---|
| Room Temp. | 2710 | 2870 | 2190 | 16 |
| 260°F | 2770 | 3060 | 2320 | 17 |
| -70°F | 2567 | 2870 | 2260 | 17 |

**Soaked at elevated temperature for 30 minutes before testing.*

### TABLE IV
**Variation of Shear Strength (Room Temperature and 260°F) With Time of Bonding After Lay-Up of Liquid Epoxy Resin Adhesive, Cured With Metaphenylene Diamine.**

All Standard Lap Shear Panels Bonded 300°F, 10 psi, 45 minutes

Panels Laid up with fresh adhesive.

| Time of Bonding After Lay-Up with Fresh Adhesive* | Shear Strength Room Temperature psi | % Cohesive Failure | 260°F psi | % Cohesive Failure |
|---|---|---|---|---|
| | Hours | Average** | | Average*** |
| 1: | 0 | 2748 | 100 | 2684 | 60 |
| 2: | 1 | 2912 | 100 | 2842 | 60 |
| 3: | 4 | 2910 | 85 | 2991 | 100 |
| 4: | 7 | 2924 | 50 | 3484 | 90 |
| 5: | 19 | 2933 | 40 | 3503 | 60 |
| 6: | 23 | 2743 | 40 | 2917 | 50 |
| 7: | 43 | 2568 | 30 | 2247 | 55 |
| 8: | 73 | 2796 | 30 | 2598 | 50 |
| 9: | 97 | 2618 | 30 | 2187 | 50 |
| 10: | 121 | 2950 | 30 | 2122 | 50 |
| 11: | 15 Days | 1670 | 0 | 1336 | 40 |
| 12: | 30 Days | 2479 | 10 | 1588 | 85 |

* Adhesive was 4-1/2 hours old before all panels were laid up.
** Average of 4 Specimens.
*** Average of 6 Specimens.

### TABLE V
**Shear Stress At Various Temperatures (Each Average is an Average of 20 Specimens)**

| Temperature | Average | Maximum | Minimum |
|---|---|---|---|
| -70°F | 2904 | 3340 | 2630 |
| Room Temp. | 3100 | 3600 | 2685 |
| 260°F | 3275 | 3750 | 2740 |
| 300°F | 1461 | 1980 | 1080 |
| 325°F | 1180 | 1390 | 950 |
| 350°F | 736 | 910 | 580 |
| 400°F | 507 | 600 | 380 |
| 500°F | 328 | 408 | 240 |

*All panels bonded 300°F, 10 psi, 45 minutes.*

# Asbestos Reinforcement for Plastics Laminates

D. V. Rosato

*Raybestos-Manhattan, Inc.*

Figure 1. Electron micrograph of Chrysotile asbestos

**Asbestos as a reinforcement for plastic laminates has certain interesting characteristics such as high modulus of elasticity, high strength at elevated temperatures and the ability to combine with other reinforcements which improves porosity resistance and certain physical properties. Various forms of asbestos are available; however, to date Chrysotile is predominantly used for reinforcing plastics. There are available for commercial use various styles in the form of felts, papers, yarns and fabrics. Also available are combinations of asbestos-glass mixtures. Preimpregnated asbestos products have been produced with satisfactory end results. Research and development are continually producing and obtaining more complete and concise data.**

ASBESTOS ROCK IS widely distributed over the crust of the earth in many forms. The most widely used are the softer varieties such as Chrysotile which is now found in abundance in Canada as well as Africa. (P. 9 of Ref. 2) Chrysotile is a hydrated silicate of magnesium. Chemical composition is as follows: (Ref. 2) $SiO_2$—37-44%; MgO—39-44%; FeO—0.0-6.0%; $Fe_2O_3$—0.1-5.0%; $Al_2O_3$—0.2-1.5%; $H_2O$—12.0-15.0%; CaO—Tr.-5.0%.

For the manufacture of reinforced plastics, long fiber asbestos is used commercially. Length of long fibers range up to ¾ inch. The shorter fibers are used principally for such items as building insulating boards, asbestos cements and asphalt tile.

The basic single fiber of Chrysotile is the thinnest and softest inorganic fiber known. It is a smooth cylinder about one fiftieth micron—or 200 angstrom units—or 1/1,250,000 inch (3)in diameter. For comparison, the very finest glass fibers measure ½ micron and the most commonly used glass fibers are in the order of 8 microns in diameter (4).

Chrysotile is found in veins of serpentine rock. It is a fibrous alteration of the constituents of serpentine as evidenced by the fact that the chemical composition is the same for both chrysotile and serpentine. The fibers run crosswise in the vein, or perpendicular to the enclosing rock. However, movements of the earth's crust in some localities have caused slippage along veins, resulting in "slip fiber" that lies in the veins at an angle from the perpendicular.

After the rock is mined, most of it is crushed and screened to free the asbestos. Later the partly opened fiber is further crushed, screened and cleaned by removal of serpentine sand, splints of Chrysotile and particles of magnetic iron ore which is associated with most Chrysotile. Additional processing through specially designed opening equipment and further screening to remove fibers too short for textile purposes are carried out before the asbestos goes through the carding operation. Carding (5) completes the opening and cleaning and arranges the fibers in a tenuous web. Pyrotex felts are made by this process.

Felts are currently manufactured in various styles. Style 9526 has the highest degree of strength and contains practically no organic matter. Style 9524 contains an open weave glass cloth. Bulk factor of these felts when impregnated with resin is approximately 3:1 when cured at 200 psi.

Various experimental thick mats are being developed. Typical is a mat which contains phenolic laminating resin and ranges in thickness from 20 to 80 mile. Bulk factor of these mat is approximately 3:2 when cured at 200 psi. Two layers of 80 mil mat will press to approximately ⅛ inch in thickness.

Asbestos papers, a unique form of asbestos fiber,

*This paper was presented at the Twelfth SPE Technical Conference.*

| Pyrotex Style | 9526 | 9343 | 9524 | 9372 | 9394 | 9517 |
|---|---|---|---|---|---|---|
| Grade of asbestos fiber | Extra Long | Long | Long* | Blend of Longs | Long & Medium | Medium & Short |
| Composition, by wt., % | | | | | | |
| Asbestos (min.) | 99 | 85 | 85 | 85 | 85 | 80 |
| Organic (max.) | 1 | 15 | 15 | 15 | 15 | 15 |
| Thickness, mil (± 10%)** | 10 | 8 & 10 | 10 & 15 | 8 & 10 | 10 | 10 |
| Weight/1000 sq. ft., lbs. (± 5%) | 22 | 14.5 & 17.0 | 21.0 & 31.5 | 14.5 & 17.0 | 17.0 | 17.0 |

Figure 2. Properties of Several Grades of Asbestos Felts

*Contains glass cloth — 50 & 33% nominal of total wt. for 10 & 15 mil styles.

**Based on the average ply of a measured stack height of 10 plies using micrometer with 5/8 inch diameter head with 300 gram head weight.

are the result of a chemical process (6) which produces dispersed single long fiber asbestos. Products made with this material have an extremely fine, smooth, uniform and long fibrous structure. Figure 1 is an electron micrograph of Chrysotile (not chemically opened) with its bundles of fasciated fibers. Chrysotile (chemically opened) as it exists in paper dispersions, because of the fineness of the fibers, is colloidal and specially adapted solutions are necessary to handle it in dispersed solutions.

Asbestos papers are available as either an all asbestos composition or in combination with various proportions of glass fiber approximately $\frac{1}{2}$ inch in length. They contain no organic fibers. Their organic content consists of residual, absorbed, oxidized, organic acid derivatives on the asbestos fibers and/or on the resin coupling agent on the glass fiber. This organic matter can be removed, if desired, by heat cleaning or through the use of suitable solvents.

Asbestos Papers

| Novabestos Style | 7401 | 7403 | 7410 | 7411 | 7412 |
|---|---|---|---|---|---|
| Composition, by wt., % | | | | | |
| Asbestos | 91-92 | 75-80 | 55-60 | 30-35 | 15-20 |
| Glass Fibers | 0 | 15-20 | 35-40 | 60-65 | 75-80 |
| Organic | 8-10 | 4-6 | 3-5 | 3-5 | 2-4 |
| **Thickness, mil** | **Approx. lbs. per 100 sq. yd.** | | | | |
| | 7401 | 7403 | 7410 | 7411 | 7412 |
| 2.5 | 4.0 | | | | |
| 5 | 8.8 | 10.0 | 8.8 | 7.0 | |
| 7 | | 15.0 | 11.0 | 9.9 | |
| 10 | | | 15.9 | 14.8 | 13.5 |
| 15 | | | | 22.3 | 20.1 |
| 20 | | | | | 23.1 |
| 25 | | | | | 28.5 |

These asbestos papers contain long fiber asbestos when compared to standard asbestos papers which are generally made of extremely short fiber asbestos which are not dispersed and contain a high percentage of impurities. Because single fibers can be dispersed, impurities common to asbestos can be readily removed. The extremely fine, single, and chemically dispersed fibers provide better packing properties than with standard fiber compositions. This becomes evident from the easy compressibility and formability of resin impregnated laminating Novabestos papers. Hemispherical shapes have been fabricated.

These papers are available in either a soft or stiff form. They should be considered wherever the relatively coarse pattern and structure of other asbestos fiber materials have been found undesirable. They may be used alone or combined with other reinforcements for surfacing or interleaving in laminates. They are compatible with all standard laminating resins. Papers containing glass fibers provide a thicker material which will absorb resins more readily.

## Water of Crystallization

Being a mineral, asbestos has great heat resistance and can be heated to high temperatures without burning. However, when it is heated the water of crystallization is removed from the crystal structure. When heated the hydroxyl radicals are removed from the crystal lattice. Figure 3 shows the weight loss of asbestos fibers with increase in temperature. The hydrous elements are completely removed at about 1,250 F and at 1,490 F the crystal structure changes from that of the serpentine (Chrysotile) structure to the olivine structure.

For the determination of the resin content of asbestos laminates, consideration has to be made for loss due to water of crystallization (pp. 21 in Ref. 1).

The relationship or effect in laminates when water of crystallization is lost is not known. However, limited tests have been conducted on asbestos laminates that have been exposed to temperatures up to 4,000 F. for short exposure (seconds to minutes) and the laminates have remained intact and retained strength and stiffness.

The specific gravity of Chrysotile is approximately 2.5. Its tensile strength is generally in the range of 100,000 to 200,000 psi. The Young's modulus of elasticity is frequently of the order of 25 x $10^6$ psi (P. 121 in Ref. 7).

The physical properties of laminates are largely dependent upon type of mold, molding pressure and the asbestos-resin ratio used. This characteristic is similar to those of other reinforcements. The flow characteristics of the laminating resin contained in the treated felts, mats or papers should be established to coincide with the molding or laminating pressure and techniques which will be employed.

All our physical tests were conducted in accordance with standards of ASTM or Federal Specification L-P-406b.

## Test Data on Asbestos Felts

Asbestos felts used to obtain high modulus of elasticity and other properties have been principally evaluated with phenolic resins. Other resins have been used; how-

Figure 3. Weight loss of asbestos fibers after reaching temperature for one hour

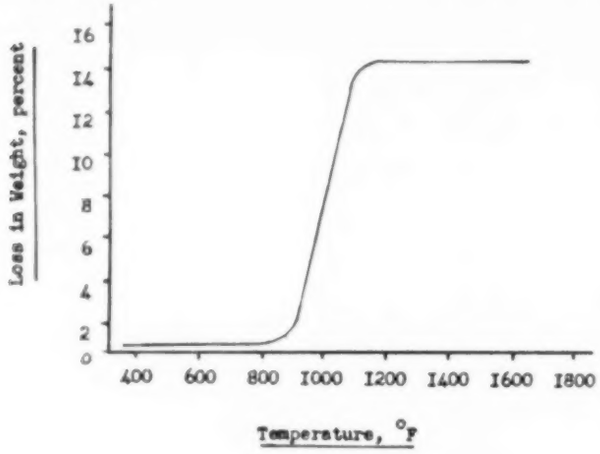
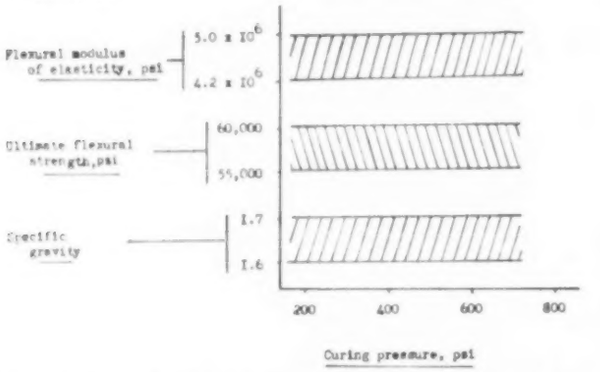
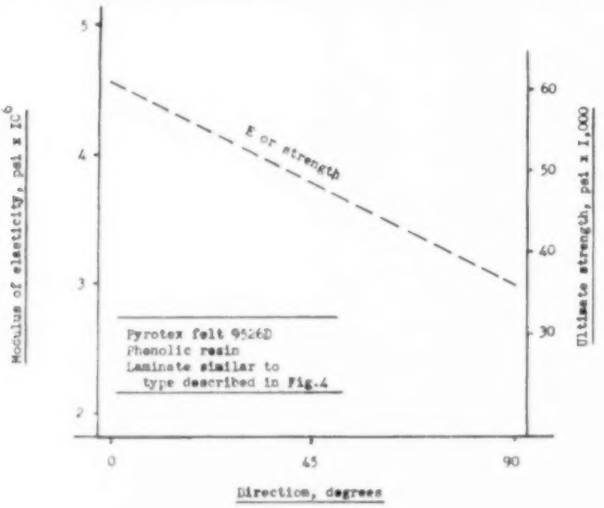



Figure 4. Properties vs. laminating pressure of asbestos felt laminates




Pyrotex felt style 9526RBI, Phenolic resin, Parallel lay up, Press & post cured, Laminate thickness 140 to 150 mils, Resin content 38 to 42 percent, Longitudinal test data (room temperature).

Figure 5. Flexural directional properties of asbestos felt laminates




ever, limited data are available. Typical data obtained on asbestos base laminates using various style felts are in Tables I, II & III, and Figures 4, 5, 6 and 7.

Asbestos base materials can be oriented in various patterns in order to produce laminates having varying directional properties. When using asbestos fabrics, an isotropic laminate can be made by laying up each asbestos sheet at least 60° to each other provided there are an equal number of laminations in each direction (8). In the case of asbestos felts or mats isotropic properties can be obtained by laying up plies only at 90° to each other. Asbestos felts or mats can be made to produce isotropic or relatively unidirectional properties.

TABLE I

Properties of asbestos felt—phenolic resin laminates

| Pyrotex Felt Style | 9526RB1 | 9517RB1 |
|---|---|---|
| **Properties at room temperature:** | | |
| Flexural properties | | |
| E x $10^6$ psi | 4.5 | 2.8 |
| Ultimate Strength, psi | 55,000 | 35,000 |
| Tensile properties | | |
| E x $10^6$ psi | 5.0 | 3.5 |
| Ultimate Strength, psi | 50,000 | 24,000 |
| Compressive properties (1) | | |
| E x $10^6$ psi | 4.3 | — |
| Ultimate Strength, psi | 30,000 | — |
| **Percent retention in properties:** | | |
| Immersion in water (30 days) | | |
| Flexural properties | | |
| E | 95 to 100 | 95 to 100 |
| Ultimate strength | 95 to 100 | 95 to 100 |
| Tensile properties | | |
| Ultimate strength | 95 to 100 | 95 to 100 |
| **Percent retention in properties:** | **9526RB1** | **9517RB1** |
| at 300°F after 200 hr. at 300°F. | | |
| Flexural properties | | |
| E | 90 | — |
| Ultimate strength | 87 | — |
| at 500°F. after 100 hr. at 500°F. | | |
| Flexural properties | | |
| E | 86 | — |
| Ultimate strength | 72 | — |
| at 700°F. after ½ hr. at 700°F. | | |
| Flexural properties | | |
| E | 75 | — |
| Ultimate strength | 60 | — |
| **Laminate history (both)** | | |
| Parallel lay up | | |
| Thickness; ⅛ inch | | |
| Specific gravity; 1.6 to 1.7 | | |
| Resin content; 37 to 40%, by wt. | | |
| Press cure; 200 psi, 30 minutes @ 300°F. | | |
| Post cure; 24 hrs. each at 250, 300 & 350°F. and 18 hrs. at 400°F. | | |
| Phenolic resin | | |
| Longitudinal test data (Ref. (1) obtained outside of R/M) | | |

TABLE II

Properties of asbestos felt—polyester resin laminates

| | | |
|---|---|---|
| Pyrotex Felt Style | — | 9517RB-3 |
| Polyester resin | | |
| Parallel layup | | |
| Press cure | — | 15 to 50 psi |
| Resin content by weight | — | 40 to 60% |
| Specific gravity | — | 1.45 to 1.55 |
| Longitudinal test data | | |
| Ultimate flexural strength, psi | — | 25,000 to 32,000 |
| Flexural modulus of elasticity, psi x $10^6$ | — | 1.6 to 2.0 |

## TABLE III

### Properties of asbestos felt—Silicone resin laminates

Dow Corning DC-2106 Silicone resin was impregnated in Pyrotex felts. Preliminary data are presented. These data were obtained from Dow Corning Corp.

Laminates were parallel layups, pressed at 300 psi for 30 minutes at 175°C, 1/8" thick, 25 plies, 45-50% resin content and 1.7 — 1.8 specific gravity.

| Pyrotex Felt Reinforcement | Cure Hours (1) at 250°C | Ultimate Flexural Room Temperature | Strength, psi 500°F. (2) |
|---|---|---|---|
| 9526-NS | 40 | 31,300 | 14,400 |
| 9529-NS (3) | 40 | 34,800 | 10,700 |
| 9526-NS (3) | 300 | 29,700 | 16,400 |
| 9529-NS (3) | 300 | 32,900 | 16,000 |

(1) Prior to heating at 250°C, the laminates were heated 16 hours at 90°C, 2 hours each at 125, 150, 175, 200 and 225°C.

(2) Tests conducted at 500°F. after one-half hour exposure in addition to the ageing listed above.

(3) Pyrotex Felt style 9529 is a modification of style 9526 through the addition of an open weave glass cloth. This construction is similar to style 9524 described in R/M Technical Bulletin T-55 (ref. 1)

To determine the effect of higher temperatures, laminates were tested after exposure of one hour each at 400, 500, 600, 700, 800, 900, and 1000°F. The ultimate flexural strength tested at room temperature was 13,400 psi.

## TABLE IV

### Properties of asbestos paper laminates

| | |
|---|---|
| Laminating resin | —Polyester or Phenolic |
| Novabestos paper style | —7411 or 7412 |
| Parallel layup | |
| Press cure | —15 to 200 psi |
| Resin content, by weight | —60 to 70% |
| Specific gravity | —1.2 to 1.5 |
| Longitudinal test data | |
| Tensile properties, psi | |
| Ultimate strength | —20,000 to 30,000 |
| Modulus of elasticity x $10^6$ | —1.5 to 2.5 |
| Flexural properties, psi | |
| Ultimate strength | —24,000 to 40,000 |
| Modulus of elasticity x $10^6$ | —1.4 to 2.0 |
| Ultimate compressive strength, psi | —24,000 to 45,000 |

## Test Data on Asbestos Papers

Asbestos papers are used to manufacture relatively thin laminates, or to act as a cushioning material through interleaving with other reinforcements. They are also used as a surfacing sheet. Test results are shown in Table IV and Figure 8.

When Novabestos papers are used to cushion parallel glass fibers, a combination is produced that provides desirable handling characteristics for mass producing parts.

## Summary

Asbestos when used as a plastic laminate reinforcement has shown that certain interesting characteristics are produced such as:

High modulus of elasticity
High tensile yield strength
Retention of strength at elevated temperatures
Acid resistance (9)
Flame resistance
Insulating and thermal properties

(Please turn to page 51)

Figure 6. Typical tensile stress-strain curve for asbestos felt laminates

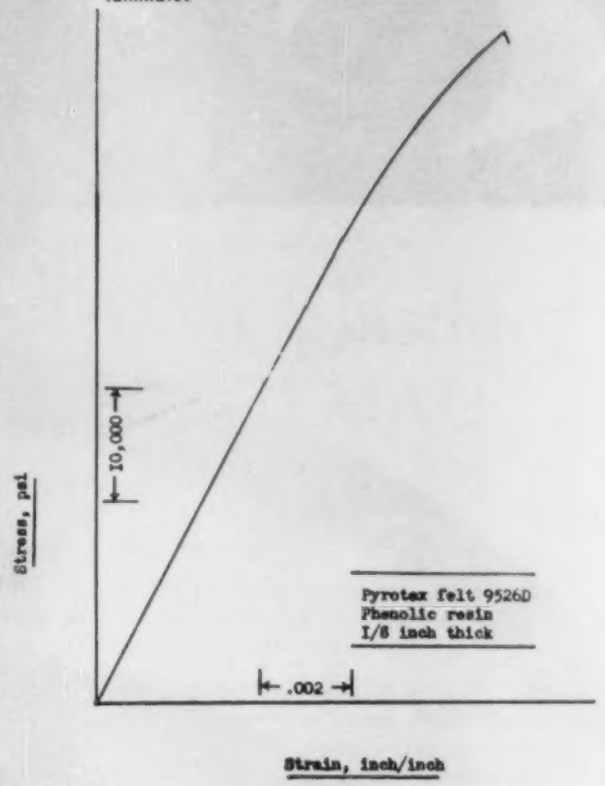
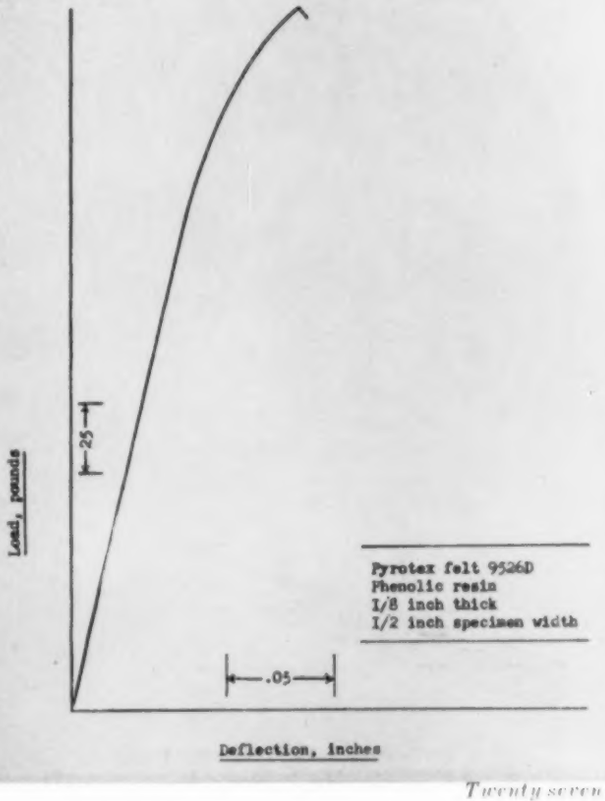



Figure 7. Typical flexural load-deformation curve for asbestos felt laminates



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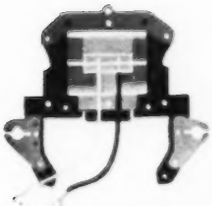
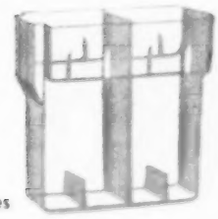
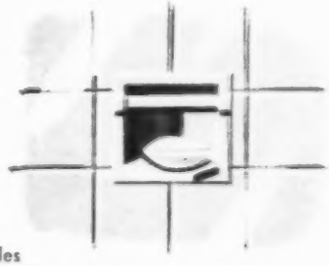


For electrical parts

For fan blades



For battery cases



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# Protective Potting of Glass Vacuum Tubes and Ceramic Capacitors

Asaf A. Benderly, J. W. Tidler and B. Greene

*Diamond Ordnance Fuze Laboratories*

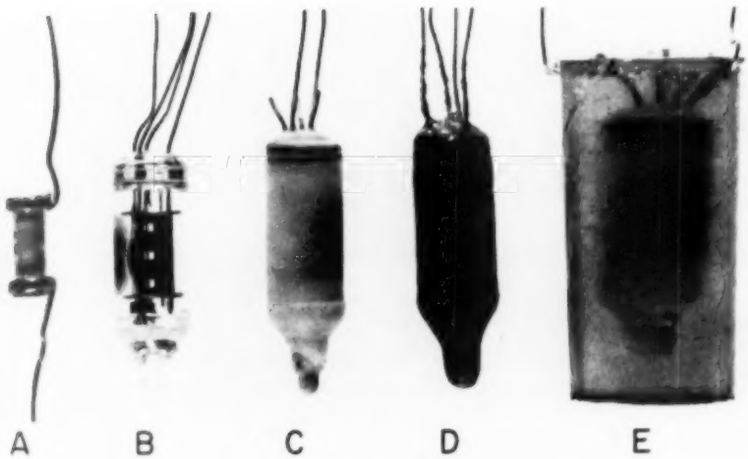



Figure 1. Ceramic capacitor and glass vacuum tubes for potting: A, bare ceramic capacitor; B, bare vacuum tube; C, vacuum tube coated with silicone rubber; D, vacuum tube coated with silicone oil and rolled in small nitrogen-filled phenolic spheres; E, potted silicone-rubber-coated vacuum tube.

THERE HAS BEEN a long history in industrial plants, as well as in government laboratories, of damage done to delicate electronic components when they are potted, or encapsulated, in thermosetting resins. Thermosetting resins are preferred for potting of fuzes in view of the elevated temperatures these assemblies frequently encounter in service. Selenium diodes, vacuum tubes, and ceramic capacitors are the components that have been the most difficult to pot without damage.

Most of the difficulties in potting selenium diodes stem from the fact that they are sensitive to exposure to high temperatures. In this laboratory it has been shown that diode back-resistance may deteriorate at temperatures as low as 80°C (176°F). Thermosetting resins require heat to cure them and, in addition, they spontaneously generate heat during the curing process. Consequently, when selenium diodes are being potted in an assembly, it is necessary to control the process so that the maximum temperature reached in the assembly, a result of the sum of the oven temperature and the exothermal heat, is below that tolerated by the diode.

The difficulty in potting glass vacuum tubes, and some ceramic components, has been mechanical cracking of the glass or ceramic as a result of differential expansion and contraction of the resin. Damage can result in any of three ways:

1. After the resin is cured and the assembly is removed from the oven, the resin contracts as a result of cooling. The component, which normally contracts less than the resin, may be crushed by the contracting resin.

2. When an assembly is temperature-cycled, the component may be further subjected to compressive forces and crushed during the cold part of the cycle. This effect can be quite severe at -65°F.

3. If the resin adheres well to the component, it is possible that the latter will be subjected to tensile stresses during the hot part (up to 165°F) of the cycling process due to the greater expansion of the resin. In some configurations the component is subjected to non-uniform stresses and it experiences shearing forces. These stresses could result in the tearing apart of the component.

The object of the work reported herein was to investigate various potting procedures, and the tube and capacitor failures that could be attributed to these procedures. Specifically, it was desired to develop one or more satisfactory procedures which would permit recommendations to be made for the potting of a particular amplifier. In general, it was expected that this study would yield information of value in the potting of various electronic assemblies.

## Experimental Methods

In each test a number of tubes, or capacitors, was treated according to the first seven steps listed below; in some cases the procedure was extended to include step 8.

1. Coated or left uncoated, as desired (see Fig. 1).
2. Tested electrically.
3. Suspended by the leads in cavities of a particular configuration.
4. Encapsulated in resin which was formulated and poured around the component, filling the cavity.
5. Assembly cured for a specific time at a specific temperature.
6. Assembly cooled to room temperature and components again tested electrically.
7. Assembly temperature-cycled and components again tested electrically.
8. Assembly mounted, fired in an air gun to subject it to a high acceleration, and components again tested electrically.

*This paper was presented to the Symposium on Casting Resins January, 1956 at the National Bureau of Standards. This article also was originally published in the July, 1956 issue of Electronic Equipment.*

The details of the above-listed steps are:

**1. Coating.**

a. Several batches of tubes were dipped in silicone oil to prevent potting resin from adhering to the tubes and cracking them in tension or shear. The film of silicone oil was permitted to dry for at least one hour before potting.

b. One batch of tubes was dipped in silicone oil and rolled in small nitrogen-filled phenolic spheres (see Fig. 1D). It was hoped that the hollow spheres would collapse under contractive forces and leave voids that would protect the tube from failure under compression.

c. Several batches of tubes were coated with a layer of room-temperature-curing silicone rubber (see Fig. 1C). This layer was 0.002 to 0.004 inches thick, and was limited by the diameter of the tube-hole in the specific catacomb. The silicone rubber was compounded by mixing together two constitutent parts, both 50 percent solids in xylene. The viscosity of the mixture was so low that it was necessary to dip and dry the tubes three times before a coating of adequate thickness was obtained. To reduce the operation to a single dip, the silicone rubber was mixed and heated for 40 minutes in an oven at 55°C (131° F). The material partially cured, and thickened to the desired viscosity, and the tubes were dipped in it. The coating on the tubes was permitted to cure further for at least 18 hours at room temperature. Later it became possible to procure the silicone-rubber constituents as 100-percent-solids materials. Therefore, in subsequent work, the dipping mixture was made up to 60 percent solids in xylene. The multiple coating or the precuring operations were thereby eliminated.

d. Several batches of tubes were not coated.

**2. Electrical Testing.**

a. The test consisted in the application of 1.5 volts to the tube-filament and the measurement of corresponding filament current. It was reasoned that a cracked tube would permit entry of air which would conduct heat from the hot filament faster than would be the case in a vacuum. Consequently, the temperature and, as a result, the resistance of the filament in a cracked tube would tend to be lower than that of a sound tube. This would result in an observably higher current being drawn at rated voltage. Only tubes that drew normal load during the first electrical test were potted. To aid the testing operation the filament leads were left long and all the other leads to the tubes were clipped short.

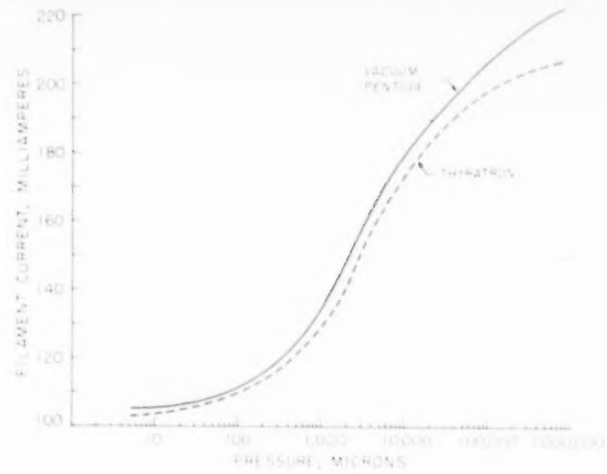
To investigate the validity of the basis for the electrical test, the relationship between tube-filament current and pressure within the tube was determined. The glass envelope of a tube was punctured and the tube was placed in the bell-jar of a vacuum system. The system was pumped down to pressures varied between 5 microns and atmospheric pressure. The filament current at each pressure was measured. The data are presented in a semi-log plot in Figure 2. It is interesting to note that fairly small leaks into the tube envelope can be detected by the measurement of the filament current. In the case of a completely potted tube, time must be allowed for air to permeate through the resin or along the tube leads. In this work it was found that testing after two weeks disclosed no more leaky tubes than after two days.

b. The capacitance of a ceramic capacitor was measured on a capacitance bridge. It was assumed that breakage or other damage to the capacitor would appear as a substantial change in the measured value of capacitance.

**3. Configurations of Potting Assemblies**

**Type a.** The tube was suspended by its leads in a greased, $\frac{5}{8}$-inch diameter, hole in a thick polystyrene block. The hole was slightly tapered to allow the resin plug with the tube imbedded therein (see Fig. 1E) to be pushed gently out of the block.

Figure 2. Electron tube-filament current at 1.5 volts and various pressures.




**Type b.** The tube was inserted, tip down, into a polystyrene catacomb, 0.6-inch deep, so that the base of the tube and the leads extended above the catacomb (see Fig. 3A). The catacomb was placed in an amplifier can and resin was poured into the can to a level about $\frac{1}{4}$-inch above the base of the tube.

**Type c-1.** The tube was inserted in polyethylene catacombs. In this case, the catacomb provided a completely covered, cylindrical chamber with a corrugated wall. The leads of the tube were brought through small holes in the cover (see Fig. 3B). The lead holes and the cover joint were coated with silicone oil to prevent resin from entering the tube chamber. The catacomb was placed in an amplifier can and resin was poured into the can to a level about $\frac{1}{4}$-inch above the top of the tube chamber.

**Type c-2.** The tubes were treated exactly as in Type c-1 but in this case one polyethylene catacomb was placed and potted in both top and bottom hollowed halves

## Table I—Potting Resin Formulations

| Resin Formulation | Constituent | Parts By Weight |
|---|---|---|
| 1 | Epoxy resin | 100 |
| | Salt of 2-ethyl hexoic acid and 2,4,6 — tri (dimethyl-aminomethyl) phenol | 10.5 |
| 2 | Epoxy resin | 100 |
| | Diethylene triamine | 10 |
| 3 | Epoxy resin | 55 |
| | Polysulfide rubber | 45 |
| | Diethylaminopropylamine | 5.5 |
| 4 | Epoxy resin | 55 |
| | Polysulfide rubber | 45 |
| | Diethylene triamine | 5.5 |
| 5 | Epoxy resin | 50 |
| | Polysulfide rubber | 50 |
| | Diethylene triamine | 5 |
| 6 | Styrene-modified polyester resin | 98 |
| | Methylethylketone peroxide | 2 |
| 7 | Wax | 80 |
| | Polyisobutylene | 20 |

of an aluminum cylinder (see Fig. 4). The two halves (one threaded on the end) were held together by screws. The walls of the hollowed cavities were undercut to provide a mechanical lock between the casting resin and the aluminum cylinder.

**Type d.** The tube was suspended by its leads in a small glass jar approximately ¾-inch in diameter.

**Type e.** A tube was suspended by its leads in each of ten ⅝-inch diameter holes machined into both the top and bottom halves of an aluminum cylinder which was threaded at one end (see Fig. 5). The two halves of the cylinder were held together by a screw. Each of the tube holes was also threaded in order to provide a mechanical lock between the casting resin and the aluminum cylinder.

**Type f.** The capacitor was inserted flat on the bottom of a polystyrene catacomb in an amplifier can. The leads protruded through the upper surface of the resin.

**1. Potting**

The desired resin was formulated in accordance with the proportions listed in Table I. The resin was then carefully poured into the assembly so as to avoid the formation of entrapped air bubbles.

Where a wax-polyisobutylene mixture was used, it was heated to 135° C (275° F). The mixture had a melting point of ca. 90° C (194° F) and was formulated in the proportions shown in Table I, Formulation No. 7. No cure was involved; the mixture was simply allowed to cool and solidify in place.

**5. Curing**

All assemblies potted in epoxy resin were cured for 18 hours at 55° C (131° F). Those potted with epoxy polysulfide-rubber copolymer were cured for 18 hours at 65° C (149° F). Those potted with polyester resin were allowed to cure for 18 hours at room temperature followed by a 2-hour cure at 65° C (149° F).

**6. Cooling**

After the cure was complete, the assembly was removed from the oven and allowed to cool to room temperature. Subsequently, the components in the assembly were subjected to the same electrical test as that described under the section entitled "Electrical Testing."

**7. Temperature Cycling**

Most of the potted and tested assemblies were exposed to ten or eleven cycles, each consisting of 8 hours at 160° F immediately followed by 16 hours at -65° F. A few batches were exposed to only two cycles. After this cycling, the components were again tested electrically. A temperature of 160° F was found to be sufficient to cause some flow of the wax-polyisobutylene mixture. Therefore, cycling of the wax-polyisobutylene plugs was restricted to seven cycles of 8 hours at room temperature followed by 16 hours at -65° F.

**8. Shock Testing**

Groups of tubes that had been coated with silicone oil, or silicone rubber, or in some cases left uncoated, and that had then been potted with various resin formulations, according to configuration types c-2 or e, were subjected to a high acceleration. The tube assemblies were threaded into a blunt-nosed, steel projectile which was fired in an air gun. This air gun consists of a steel tube, whose inside diameter is 4 inches and whose length is 96 feet, connected to a 70-cu.-ft. tank which supplies the compressed air needed for acceleration of the projectile. The air tank is equipped with a quick-release valve. The catch-box of the air gun is provided with lead blocks to stop the projectile suddenly and produce the high accelerative force. The acceleration attained by each round was measured with an accelerometer whose operation depends on the extent of deformation of a copper sphere.

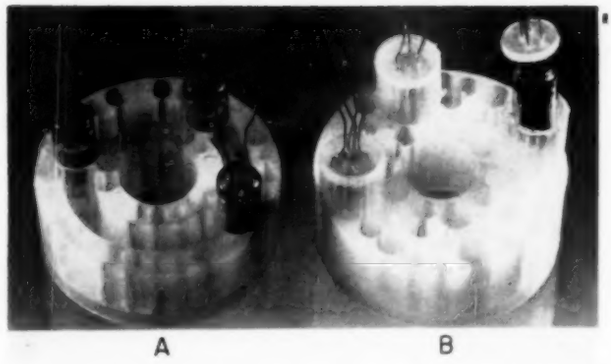



Figure 3 Vacuum tubes in amplifier catacombs: A, short polystyrene catacombs; B, polyethylene catacombs with resin-tight tube chambers.

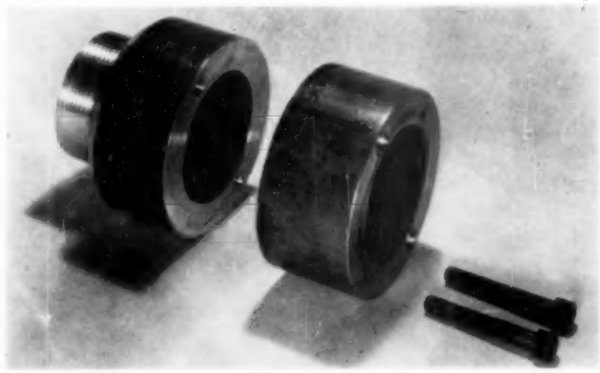


Figure 4 Aluminum shock-testing housing in which are potted polyethylene catacombs containing vacuum tubes.

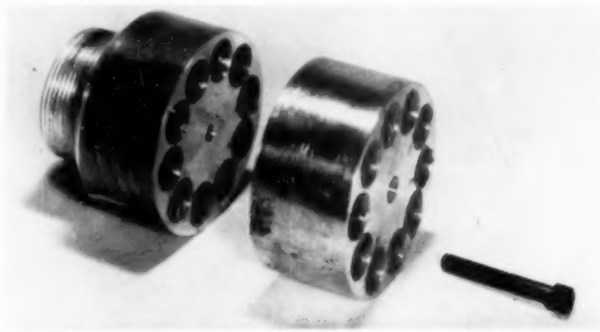


Figure 5 Aluminum shock-testing housing in which coated and uncoated vacuum tubes are potted.

After the firing of each round, the assembly was removed from the projectile, disassembled, and tested electrically.

## Discussion

Test results are listed in Table II and may be summarized as follows:

1. No ceramic capacitors cracked as a result of potting or temperature cycling.

2. Between 10 and 80 percent of vacuum tubes, uncoated, coated only with silicone oil, or coated with silicone oil and small nitrogen-filled phenolic spheres, and then potted in epoxy resin, cracked after potting or after temperature cycling.

3. More than half of the tubes potted directly in an

## Table II—Effect of Potting, Temperature Cycling, and Shock-Testing on Components

| Test No. | Number of Components Tested | Coating | Potting Formulation (See Table I) | Type of Configuration of Potting Assembly (See Experimental Methods) | Failures Resulting from Potting | No. of Temperature Cycles at -65 to +160°F | Failures Resulting from Cycling | Average Air Gun Acceleration (1000 g's) | Failures Resulting from Acceleration | Total Failures |
|---|---|---|---|---|---|---|---|---|---|---|
| Tubes: | | | | | | | | | | |
| 1 | 100 | None | 2 | e | 70 | 1 | 18 | — | — | 88 |
| 2 | 100 | None | 1 | e | 38 | 10 | 40 | 17 | 14 (6,7) | 92 |
| 3 | 80 | None | 1 | e | 36 | — | — | 16 | 15 (7) | 51 |
| 4 | 122 | None | 1 | e-1 | 0 | 2 | 0 | — | — | 0 |
| 5 | 58 | None | 1 | e-1 | 0 | 11 | 0 | — | — | 0 |
| 6 | 96 | None | 1 | e-2 | 0 | 10 | 11 (1) | 16 | 18 (6,7) | 29 |
| 7 | 99 | Silicone Oil | 1 | a | 7 | 2 | 4 | — | — | 11 |
| 8 | 129 | Silicone Oil | 1 | b | 6 | 2 | 7 | — | — | 13 |
| 9 | 114 | Silicone Oil plus Spheres | 1 | a | 1 | 2 | 15 | — | — | 16 |
| 10 | 198 | Silicone Rubber | 1 | a | 1 | 11 | 4 | — | — | 5 |
| 11 | 96 | Silicone Rubber | 1 | b | 0 | 11 | 1 | — | — | 1 |
| 12 | 100 | Silicone Rubber | 1 | e | 0 | 10 | 1 | 17.5 | 14 (6,7) | 15 |
| 13 | 296 | None | 6 | a | 0 | 11 | 8 | — | — | 8 |
| 14 | 100 | None | 6 | e | 0 | 10 | 19 | 14.5 | 30 (6,7) | 49 |
| 15 | 100 | None | 7 | a | 0 | 9 (4) | 1 | — | — | 1 |
| 16 | 50 | None | 3 | d | 0 | 2 | 25 (3) | — | — | 25 |
| 17 | 100 | None | 5 | e | 0 | 10 | 99 | — | — | 99 |
| 18 | 100 | Silicone Oil | 3 | a | 1 (2) | 11 | 0 | — | — | 1 |
| 19 | 100 | Silicone Oil | 4 | e | 0 | 10 | 3 | 18.5 | 34 (6,7) | 37 |
| **Ceramic Capacitors:** | | | | | 0 | 2 | 0 | — | — | 0 |
| 20 | 36 | None | 1 | f | 0 | 2 | 0 | — | — | 0 |
| 21 | 36 | Silicone Oil | 1 | f | | | | | | |
| **Complete Amplifiers:** | | | | | 0 | 2 | 8 (5) | — | — | 8 |
| 22 | 43 | Silicone Oil | 1 | b | | | | | | |

1. These 11 failures were attributed to leakage of resin into what should have been resin-tight chambers. No cycling failures resulted among the remaining 85 tubes where the polyethylene chambers served their proper function of excluding resin.
2. Failure was due to an open filament, not to breakage of the glass envelope.
3. Many jars, as well as tubes, cracked. This cracking was attributed to absence of a release agent on the inside of the jar or on the outside of the tube.
4. These cycles were carried out between -65°F and room temperature because of the tendency of low-melting fractions of the wax-polyisobutylene mixture to flow at 160°F.
5. Five of these failures were traced to tube breakage.
6. Twenty tubes in each category were subjected to 10,000 g's without a single failure. Consequently, all the tubes that survived potting and temperature cycling were subjected to accelerative forces averaging between 14,500 and 18,500 g's.
7. Approximately half the tube failures resulting from accelerative forces were clear-cut cases of envelope breakage. The other half appeared as open filament circuits thereby precluding the measurement of filament current which, in other cases, served as a criterion of envelope condition.

epoxy polysulfide-rubber copolymer without the use of a release agent cracked during temperature cycling.

**4.** Three to nineteen percent of the uncoated tubes potted in a flexible polyester resin cracked during temperature cycling.

**5.** Three categories of tubes survived both potting and temperature cycling reasonably well:

a. Silicone-rubber-coated tubes potted in epoxy resin (less than 2 percent failures).

b. Tubes coated with silicone oil and potted in an epoxy polysulfide-rubber copolymer (less than 2 percent failures).

c. Uncoated tubes potted in a wax-polyisobutylene mixture (1 percent failures).

**6.** All the tubes inserted in resin-tight tube chambers which were integral parts of polyethylene catacombs, and potted with epoxy resin, survived both potting and temperature cycling.

**7.** A representative quantity of those tubes, described above in groups 2, 4, 5a, 5b, and 6, that survived potting

(Please turn to page 52)

# A Method for Evaluating . . .

# Low Temperature Shock Resistance

L. S. Buchoff

*Westinghouse Electric Corp.*

IN RECENT YEARS potted electrical and electronic circuits have been made in ever increasing numbers. In military applications such as guided missiles and aircraft, these circuits must be instantly ready to perform their job. Potting protects the units from damage by moisture, vibration, and variation in temperature and pressure. Fine wires and delicate components are particularly subject to damage from cracking of the casting material during temperature cycling. Inserts with thermal expansivity differing from that of the plastic increases this possibility. Fillers are used to reduce expansivities and plasticizers to lower the brittleness temperature.

A method of evaluation is needed to determine the effectiveness of these materials. The most obvious test is potting groups of the units in various formulations and shocking them through the temperature range they would meet in practice. The units that come through intact presumably contain the best potting material. This test is impractical in most cases, at least for testing large numbers of materials. The number of units necessary and time of testing would make the cost prohibitive. A quick reproducible test is necessary to compare the cold crack resistance of resins around embedments.

The military uses the hex bar test to a great extent. In this test a mild steel hex bar 1-inch long and ¾" across is cast in the resin to be evaluated. The hex bar, casting mold and centering cap are shown in Figure 1. Groups of specimens cast in various formulations are thermally cycled a number of times. Frequently used cycles are 85°C to −40°C, and 85°C to −65°C. After the cycling, the specimens are examined for cracks. The material showing fewest cracked specimens is considered to be the most resistant to thermal shock.

A large number of specimens are necessary to even rank of the materials. The arbitrary hex bar shape merely provides a standard stress condition. It does not represent any typical shape insert. Because this test is so widely used it was made the basis of the test described in this paper. The hex bar test at best gives an evaluation of resins only between two fixed temperatures. It appeared that a much better standard of resin performance would be the fracture temperature when the hex bar specimen is cooled at a specific rate. A method was needed to determine the cooling curve and temperature at break without affecting the results. A thermocouple imbedded in the plastic could be used to determine temperature but this would be only at one point and the bead would cause a high stress at that point. The method finally used is shown in Figure 2. The hex bar is wrapped with AWG #40 insulated wire and potted. This wire has a diameter of 0.0031 inches.

The average temperature of the specimen can be found from the resistance of the wire, as the resistivity of copper is directly proportional to its temperature over the range we are interested in. We have in effect a simple resistance

Figure 1. Apparatus for the hexagonal bar testing of resins.

Figure 2. Apparatus for extending the hexagonal bar test to include complete cooling curve and temperature at break.

thermometer. Cracking of the resin breaks the fine wire on the hex bar causing an open circuit.

In practice AWG #18 wires are run from the specimen to a Wheatstone bridge. The specimen is put in a cold box. Resistance readings are taken every minute or half minute until the wire breaks as shown by infinite resistance. The last reading before this is considered the crack temperature. The entire cooling curve can be easily calculated from the resistance readings.

Initial specimens were cast from epoxy resin catalysed with 6% piperidine and cured for 24 hours at 60°C. They were cooled in a box at −65°C. Using a constant amount of insulation, 10 test pieces broke between −28°C and −23°C in 28 to 31 minutes. In three runs the specimens were observed and noted to crack at the same time that infinite resistance was noted. Specimens having heavier insulation cooled more slowly in the 65°C ambient. These cracked at temperatures lower than the lighter covered pieces. Thus rate of cooling can be correlated with fracture temperature.

Several specimens were cast in epoxy resin catalysed with 8% diethylenetriamine and cured for 4 hours at 60°C. In many of the test runs, the resin would crack without causing discontinuity. It was observed that the cracks were hairlines compared to the much wider cracks in the piperidine catalysed units. In order to test the DET catalysed epoxy casting a path of silver conductive paint on the outside of the unit was substituted for the wire. Two possible configurations are shown in Figure 3. The resistance of this material was found to be directly proportional to the temperature. In all cases tried including filler and other catalysts, the painted path showed open at the time cracking was observed. In several tests, both the wire and painted path were used. The temperatures on the outside of the specimen was within the experimental error of the internal temperature. By using both wire and paint the extent and size of cracking could be determined on some specimens.

The test as described requires the constant services of a technician to collect the resistance time data. At best one man can handle only two simultaneous runs.

Thus the next step was automatic recording of time-temperature curve and temperature of casting cracking. This is accomplished with a twelve point recording potentiometer to monitor 12 runs simultaneously. The potentiometer measures the voltage drop across the wired units which is proportional to the resistance of the wire and therefore proportional to the temperature. A gaseous discharge tube source maintains a constant voltage of 5 volts across the network through a load change of 100 to 600 milliamps. The resistances were arranged so that this was maintained regardless of the number of paths intact. The variable resistors were several orders of magnitude higher than that of the wire in the units. Therefore an essentially constant total resistance is maintained in each branch and the currents remain unchanged. The voltage drop is truly proportional to the temperature. The potentiometer used had only a scale of 0 to 5 millivolts. Since the extrapolated resistance-temperature curve of copper shows zero resistance at −234°C and we are operating over a range of only 35°C to −65°C we would be using only about one third of the chart at most. To spread the useful temperature range over the entire chart the variable and fixed resistance branch was added to the circuit. This subtracts a constant adjustable number of millivolts from each reading. For example, if the drop across a unit varies from 25 to 21 millivolts during a run, and 20 millivolts

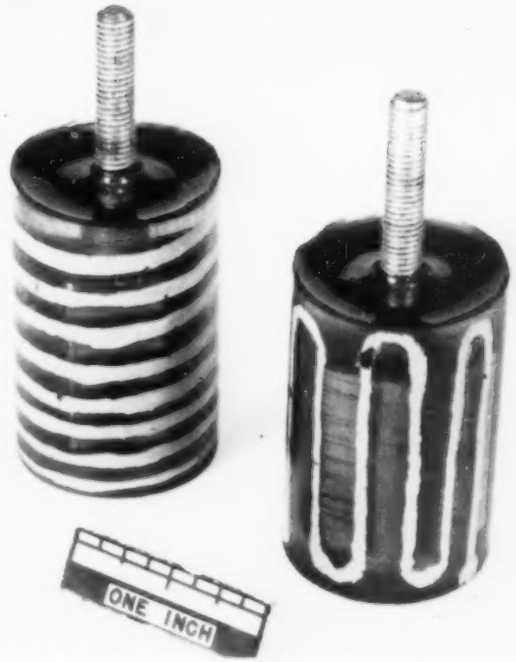



Figure 3. Specimens for test, using a conductive silver paint in place of wire.

are subtracted, the potentiometer reads 1-5 millivolts. Adjustment of the variable resistances shifted the scale and made it possible to use either the wires or the painted silver paths. By using two subtracting branches both measuring means can be used simultaneously.

In use, the potentiometer scale is marked in temperatures rather than millivolts. As each unit's wiring has a resistance differing somewhat from the other, the variable resistors in series with each unit are necessary to calibrate the instrument. It is only necessary to adjust the resistors before the test so that the potentiometer indicates the ambient temperature. The units are then put in the cold box. The temperature of each specimen is then recorded at regular intervals. When the specimen reaches its fracture temperature the resin cracks, breaking wires. The potentiometer then sees a voltage drop several times that across the specimen. The next temperature recording and subsequent points of this specimen are off scale. The discontinuity of readings at the breaking temperature is easily seen. This test was designed primarily to help evaluate casting compositions.

Because of the complex nature of the casting cracking, one test can only give a rough approximation of its cause and curves. One of the most effective ways of reducing damage to components during thermal shock is matching the expansivities of the resin and inserts. If the inserts are of only one material and the resin is matched over the entire temperature range, there should be no problem. If the materials aren't matched perfectly, the shape and size of the insert and casting will affect the resistance to cracking. The thicker the casting, the more important the temperature gradient across it and the rate of temperature change. Knowing the temperatures of shocking alone will not be enough to get reliable, reproducible data. By using a wire inside and a painted circuit

(Please turn to page 52)

# Cellulose Propionate Molding Compounds

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*Celanese Corp. of America*

CELLULOSE PROPIONATE IS manufactured by processing a hard granular flake with plasticizers. The flake used is produced by treating chemical grade cotton with a mixture of propionic anhydride and propionic acid for esterification. The quantity and type of plasticizers used regulate the flow of material in molding and constitute an integral part of the physical properties of the various formulations. These plasticizers constitute a physical mixture with the flake and enable the injection molding of the material by acting as lubricants. The same plasticizers through rigid control of type and quantity enable the manufacturer to tailor-make physical properties to suit the most exacting demands of the product.

## Physical Properties

Despite the ability of the supplier to control physical properties by material composition, the molder exerts considerable influence on molded part quality by machine operating techniques. In all cases, it is a requirement that the material be sufficiently plasticized in the molding machine and that molding temperatures be considerably above cold point operation* (minimum temperature required to fill a die). In some instances, impact strength can be doubled by a 50°F increase in machine cylinder temperatures.** The ability of a material to weld around pins or inserts is often a controlling factor even though the physical properties of the same material in a straight molding may be excellent. Cellulose propionate does not allow the molder to relax completely his vigil on machine control for quality molding, but it does offer him a material somewhat less critical to mold. The welding properties of propionate are exceptionally good which means maximum weld strength and practically invisible weld lines. The molder has approximately 70°F operating range within which he can produce parts with optimum physical properties. This range will vary somewhat, depending on equipment limitation, but in general it enables this wide temperature variation from minimum to the point just short of discoloration or breakdown.

With this background in mind, let us investigate the exact physical properties of cellulose propionate. To establish its range with relation to an established material, Figure number one shows a comparison of this material with cellulose acetate. The propionate curve is considerably above that of acetate, giving high readings of impact, 9 to 11 foot/pounds, with corresponding high flexural strength, 5500 to 6500 p.s.i. Table number one lists the physical properties of three typical cellulose propionate formulae. In reviewing this data, it is noted that the keynote is a "balance of properties." For each specific value, there can no doubt be found a material that has a slight edge, but no other material offers such a balance in considering the level of all physical properties. Cellulose propionate offers a combination of great toughness, good dimensional stability, surface hardness, and adequate resistance to heat distortion.

## General Properties

The balance of properties does not stop with this outline of specific physical data. In considering the acceptance of propionate, one must investigate its advantages in many other categories.

1. **Odor**—Propionate is free of objectionable odor.
2. **Compatibility**—Propionate is compatible with the mixed ester cellulose acetate butyrate, but incompatible with all other plastics.
3. **Fabrication**—Propionate is readily fabricated even in hard flows. Such operations as slotting, threading, piercing, riveting, and drilling have been done successfully on even H-2 flow materials.
4. **Lacquering**—The low level of plasticizer in propionate enables the use of cellulose nitrate and/or acrylate base lacquers. In this regard, it should be noted that the ability to mold essentially strain-free parts eliminates problems of skinning and fractures when utilizing solvent thinners for spray painting. Plasticizer migration is virtually ruled out in hard flows.
5. **Metallizing**—In this study, the low plasticizer level once again minimizes problems of degassing often associated with cellulosics.
6. **Cementing**—There are a number of solvents, covering a wide range of evaporating rates that can be used for cementing propionate to itself or to cellulose acetate butyrate.
7. **Surface Gloss**—Molded part finish when utilizing propionate is extremely good. In most cases, the finish obtained is the best among the cellulosics. This desirable gloss is maintained over widely varying injection cylinder temperatures and machine cycles.

## Molding

Cellulose propionate injection molds with ease on relatively short cycles. Recommendations call for the use of high pressure, fast injection speeds, but while these are desirable, they are not essential. In handling this material a pre-drying period of 2-3 hours at 160-180°F is recommended to remove moisture, since it is a cellulosic. This pre-drying period serves also as pre-heating and enables more efficient molding of the cellulosics through better cycles and lower machine temperature requirements. The

*This paper was presented at the Twelfth SPE Technical Conference.*

*Celanese New Product Bulletin NP-16 "Forticel", June 30, 1955.
**"Cellulosics"—Modern Plastics Encyclopedia, 1955.

mold shrinkage is .002-.004 inches per inch. This low factor allows the use of standard cellulosic mold design and does not necessitate new tooling. True pin point gating is not always recommended for any cellulosic but optimum results have been obtained using restricted gating techniques.* In special applications, such as multi-cavity pen and pencil molds, successful operation has been found however, with gate sizes as small as .025" using hard flow materials.

Cellulose propionate exhibits a high ASTM flow temperature but this should not be mistaken as a need for excessive temperature requirements in injection molding. The majority of formulations are found in the H to H-6 category based on ASTM flow ratings. Based on this classification, this material will injection mold at temperatures equivalent to that for a comparable cellulose acetate or cellulose acetate butyrate formula, approximately four steps softer. It is because of this that flow designations given for cellulose propionate are selected to represent actual injection molding conditions and not ASTM flow. It can be assumed that the ASTM flow is approximately four steps harder. The explanation for this characteristic is found in the viscosity behavior of cellulose propionate. The initial drop in viscosity with heat application is not as pronounced with cellulose propionate as with cellulose acetate or cellulose acetate butyrate. In the range of 290-330°F, the range of ASTM flow determinations, propionate viscosity remains relatively high and gives a high ASTM flow. As we increase temperature to the range of injection molding, 420-500°F, we find that viscosity has fallen rather rapidly and the injection molding characteristics are those of a considerably softer material. Figure number two shows the relationship of viscosity change or cellulose acetate and cellulose propionate with increase in temperature.

In reviewing the chart we note a basic straight line relationship in plotting material viscosity against heat for various flows of cellulose acetate. The selected flows represent MH through H-4 material. In ASTM classification there is a plus or minus 5°C, approximately plus or minus

*) *"Balanced Gating"—Modern Plastics, December 1954.*

Figure 1. Comparison of Cellulose Acetate and Cellulose Propionate Impact Strength

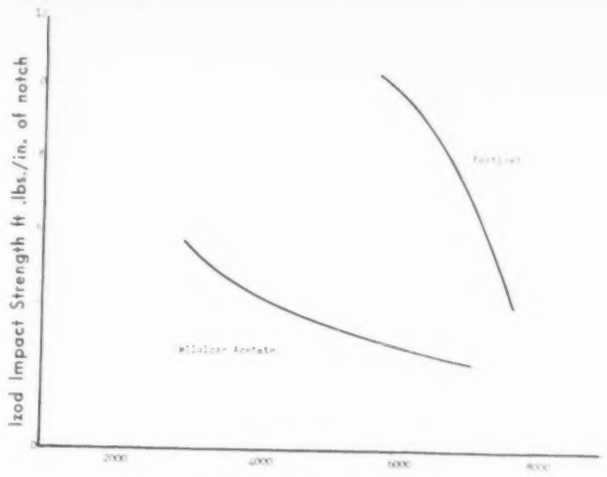
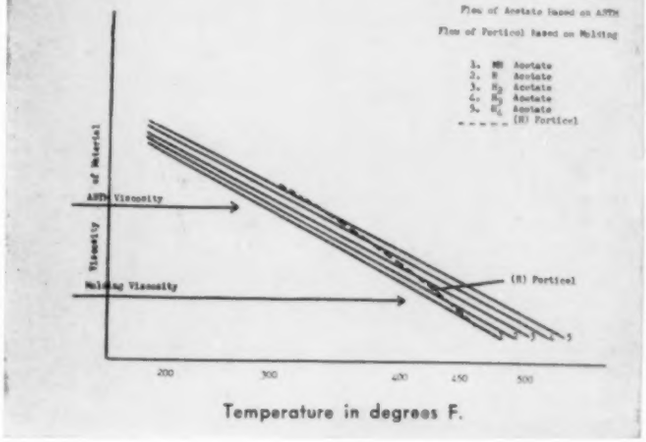



Figure 2




Cellulose Propionate Molding Compounds

TABLE I Typical Physical Properties

| Designation | ASTM Test Methods | Typical Formulas JLB-(H) | JMB-(M) | JMA-(S) |
|---|---|---|---|---|
| Flow Temperature (°C.) (A.S.T.M.)* | D569-48 | 178 | 167 | 149.5 |
| Specific Gravity | D176-42T | 1.21 | 1.18 | |
| Tensile Properties: Yield (p.s.i.) | D638-52T | 5020 | 3380 | 2410 |
| Break (p.s.i.) | " | 5240 | 3470 | 1850 |
| Elongation (%) | " | 56 | 57 | 49.6 |
| Flexural Properties: Flexural Strength (p.s.i. at break) | D790-49T | 8500 | 6400 | 4800 |
| Flexural Modulus ($10^5$ p.s.i.) | " | .30 | .23 | .17 |
| Rockwell Hardness (R Scale) | D785-51 | 94 | 62 | 12 |
| Isod Impact (ft.lb./in. notch) | D256-43T | 2.7 | 9.0 | 11.0 |
| Heat Distortion (°C.) | D648-45T | 70 | 59 | 60 |
| Water Absorption — % Sol. Lost | D570-42 | 0 | .02 | .12 |
| % Moisture Gain | " | 1.8 | 1.6 | 1.51 |
| % Water Absorption | " | 1.8 | 1.6 | 1.56 |

*)—*Cellulose Propionate material exhibits a high ASTM flow temperature which should not be mistaken as a need for excessive temperature requirements in injection molding. On the basis of ASTM flow readings, cellulose propionate will injection mold at temperatures equivalent to a comparable cellulose acetate or cellulose acetate butyrate formula, approximately four (4) steps softer. For this reason, the formula and flow designation given in II for a cellulose propionate is selected to represent actual injection molding conditions and not ASTM flow. It can be assumed that the ASTM flow is approximately four (4) steps harder.*

10°F, variance between flows. Actually the hard side of an H flow can overlap the soft side of an H-2 flow molding powder. As we project this into the 450°F. range we can establish approximately 10-15°F difference in molding temperature requirements between flow steps. The position or propionate with regard to the other cellulose esters is plotted on the graph as a broken line. The selection of a single formula and flow was made to show its relation in regard to ASTM flow and practical flow. At a viscosity sufficiently low to satisfy ASTM flow the given propionate formula requires enough heat to classify it as H-4 in this system. As we follow the continued effect of temperature on viscosity we note a considerably more rapid drop and in the range of machine operating temperatures the propionate molds with the requirements of only an H flow. One of the basic reasons for flow identification of materials is to indicate to the molder the approximate machine operating conditions to be employed. To satisfy this point it becomes necessary to deviate from ASTM standard designated meaning for flow. The decision was made to identify materials, in regard to flow, on practical molding temperature requirements. In setting up this system the flow letter was used but it is always placed in parenthesis when associated with cellulose propionate.

The formula and flow series established for this new material is quite unique in another manner. Within each flow step of a formula series is found an "A" and a "B" material. The control of plasticizer level in propionate, as compounded by Celanese, is precise enough to allow two materials with a flow step, one on the hard side of the flow and one on the soft side of the flow. A system of this type is found in no other material and allows formula selection to satisfy the most critical and exact application. Let us consider just one series offered and see what it means with regard to available molding powder formulations.

| Formula | Impact Strength ft. lbs./in. Notch | Flexural p.s.i. Break | Tensile p.s.i. Yield |
|---|---|---|---|
| JLA-(H2) | 2.25 | 8800 | 5300 |
| JLB-(H2) | 2.7 | 8500 | 5020 |
| JLA-(H) | 3.5 | 7700 | 4600 |
| JLB-(H) | 4.7 | 7200 | 4230 |

These four materials in two flow steps offer a range of physical properties at only small changes in machine temperature requirements.

Table two is given to outline the relationships that exist among cellulose acetate, cellulose acetate butyrate and cellulose propionate with regard to supplier flow designation, ASTM flow, and approximate injection molding temperature requirements. In this outline we note that a very slight temperature increase is recommended for propionate on a designated flow for flow basis. For all practical purposes, however, these materials can be considered quite equal in regard to temperature requirements.

**TABLE II**

**The following table is offered as a guide in the use of cellulose propionate for injection molding.**

| Basic Groups | Flow Designation CA-CAB* | Flow Designation CP | ASTM Flow °C CA-CAB | ASTM Flow °C CP | Injection Molding Temp. CA-CAB | Injection Molding Temp. CP |
|---|---|---|---|---|---|---|
| Hard | H | (H) | H | H | 400 | 410 |
| Medium | M | (M) | M | H | 380 | 390 |
| Soft | $S_1$ | ($S_1$) | $S_1$ | MH | 350 | 360 |

**These are typical compositions which would fall in each group. For each flow step, cellulose propionate molding composition will vary 10-15°F.*

*Code: CA —Cellulose Acetate*
*CP —Cellulose Propionate*
*CAB —Cellulose Acetate Butyrate*

## Compression Molding

Initial field trials have been made with propionate on compression molding applications. Tests show that the high viscosity characteristics at lower temperatures do limit the material in this field. A designated (M) formula was unsatisfactory at 340°F on a 12 minute cycle and indications are that only the softer flows can be used. This limitation would of course be offset if considerably higher temperatures were available for a compression operation. Continued study and experimentation is being carried out.

## Extrusion

Limited field trials in extrusion of pen tubes, pipes, and the like, have shown that there are no basic problems in the handling of propionate in commercial machines. The operator has been able to control part dimensions readily and has found no sacrifice in machine output. The sole factor which may curtail extensive development in this field is the slight gel surface of the extruded product. Reports will be forthcoming concerning the extrusion characteristics of cellulose propionate upon completion of field work now under way.

## External Pressure Vessels . . .

(Continued from page 19)

rate was 0.0000235 grams per 24 hours per square inch of exposed area per ¼" wall thickness. When the pressure was increased to 600 psi, the average rate was measured at 0.0000232.

With the above data, useable criteria are now available for estimating external pressure vessels. By selection of the proper glass reinforcements, wall thickness and effective lengths, as controlled by ribbing or overall length of the vessel, designs can be readily produced. The long-term loading data indicate that these vessels may be continuously loaded up to 50% of their short-term collapsing pressure resistance and can be expected to function for at least one year. Glass reinforced plastics may, therefore, be considered as very suitable materials of construction for specialized external pressure vessel applications.

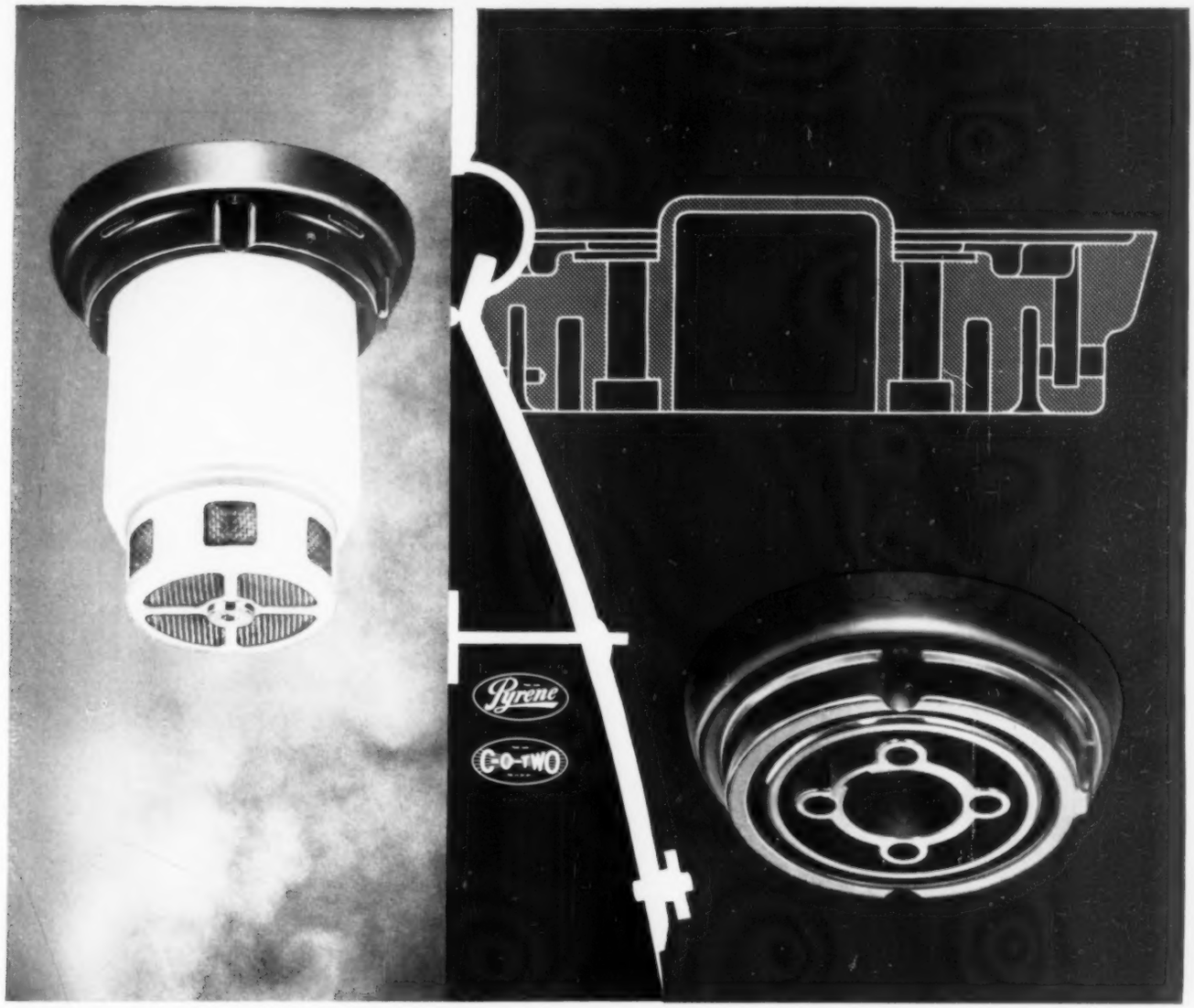
## References

(a) "Reinforced Plastics Tubing" by F. Robert Barnet and H. B. Atkinson, Jr., **SPE Journal**, Oct. 1953, p. 22.

*"Fifteen Years of Plastics Progress"*

THE THIRTEENTH ANNUAL TECHNICAL CONFERENCE OF THE SOCIETY OF PLASTICS ENGINEERS

St. Louis January, 1957




## it's DURITE for the new Pyrene—C-O-Two fire guard

This remarkable fire protection device, approved by Underwriters' Laboratories, Inc. utilizes radio-active material to sense the combustion gases and smoke which precede the outbreak of flame. The compact C-O-Two Pre-Detector Heads are fastened to the ceiling in strategic locations throughout a building and must be trouble free over a long service life. Since accurate assembly is vital to perfect operation, key molded parts must have and retain a high degree of dimensional stability.

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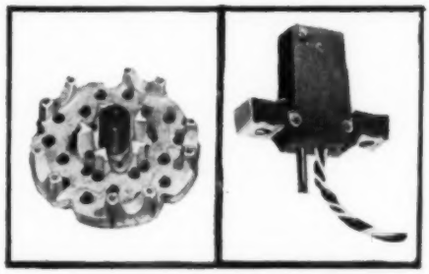

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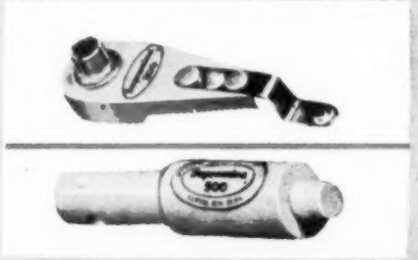
**TYPICAL APPLICATIONS**



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**"The objects of the Society shall be to promote in all lawful ways the Arts, Sciences, and Engineering Practices and Standards connected with the utilization of plastics."**

## How to Get Ahead

What everybody wants to know is how to get ahead—both in finances and prestige. To help you do both is the main reason for the existence of the SPE. There really isn't any such thing as the plastics industry, there are only individual men engaged in making and selling plastics, and it is toward those individual men that SPE is oriented.

You get ahead by know-how, personal contacts and public acceptance of your products. The SPE helps with the first two with technical meetings, the Annual Conference and the SPE Journal.

The 13th Annual Conference will be in St. Louis in January 1957. News from St. Louis is exciting. The number of excellent papers already committed to be presented provides an embarrassment of riches for the program committee. The theme of the Conference is "Fifteen Years of Plastics Progress." An innovation will be improved registration procedures, and the provision of an Information desk to help with all problems of registrants. The Information desk will be staffed with National Councilmen who will also extend an attentive and authoritative ear to complaints and to suggestions.

The local Section Conferences this year have also established an important service; New York and Newark this Spring on Materials for Packaging, and Rochester a day long seminar with eight papers presented. Coming on September 20 is an ambitious and interesting program on plastic pipe with four panel discussions, at the Sheraton Mount Royal Hotel in Montreal, Quebec.

The SPE Journal has grown in size along with the SPE itself. Biggest innovation is publication of issues in July and August, for the first time.

All this has something real to do with how you get ahead, not only in the obvious ways of providing know-how and personal contacts, but also in the public acceptance which follows on the prestige of an industry's engineering organizations.

Active membership in an active organization has always been one of the requisites for real professional advancement.

## Society of Plastics Engineers, Regional Technical Conference On Plastic Pipe

**Thursday, September 20, 1956**
**Sheraton Mount Royal Hotel,**
**Montreal, Quebec, Canada.**

The theme of this conference will be "Applications Development," with our efforts concerted in an attempt not to review the production problems arising in the manufacture of plastic pipe but rather a thorough discussion on the varied end uses for pipe and possible new applications which are being considered in the various phases of industry.

The program will consist of 4 panels, a luncheon and a cocktail party for a total cost of $10.00.

Raw Materials Panel (9 A.M.)

Moderator—Dr. R. V. V. Nichols—McGill University, Montreal. Speakers have been obtained from each raw material producer and they will discuss the advantages of polyethylene, cellulose acetate butyrate, rigid PVC, Kralastic and reinforced.

**Oil, Gas and Petroleum Panel**

Panel members will be obtained to secure information on artificial and natural gas distribution in cities, oil field handling and several other phases which would apply to this category.

Following these panel discussions, a luncheon will be held in the hotel and afternoon sessions will commence at approximately 2:00 o'clock P.M.

**Municipal and Civil Defense Applications Panel**

Moderator—Mr. J. E. Hanna—National Research Council, Ottawa, Ontario. Panel members are:

Dr. W. Tiedeman of the National Sanitation Foundation, University of Michigan; Dr. A. E. Berry, Director of Sanitation Engineering, Department of Health; Col. P. Cawdron, Federal Civil Defense Coordinator, Department of National Health & Welfare; Mr. W. Ruddock, Beardmore & Company Limited (Pipe Producer).

A state sanitation engineer will also be added to this panel. Our last panel in the afternoon will be:

**General Industrial Corrosive Service**

Moderator—M. W. Keddie, Renfrew Aircraft and Engineering Company. Panel members have been obtained from the Ontario Hydro, DuPont Company, Canadian International Paper Company, National Tubing Company of Pittsburgh and an additional speaker will be obtained representing textiles or the refining industry.

A cocktail party will be held at approximately 5:00 P.M. in the hotel on completion of this final panel.

Registration fees are $10.00 per person which include admission to both morning and afternoon panels, luncheon and participation in the cocktail party during the early evening.

Pipe Conference committee members are:

Conference Chairman, D. F. Mason, Canadian Industries Limited, Vice-Chairman, R. A. Noble, Canadian Industries Limited; Treasurer, J. Powers, Ontario Steel Products Company; Secretary, J. C. Gillis, Northern Electric Company Limited; Publicity, O. G. Ball, Canadian Chemical Company; Program, E. Littlejohn, Bakelite Company; Registration, M. E. Morrison, Industrial Plastic Extrusions Company; Entertainment, C. K. Dewar, Dominion Rubber Company; Hotel Arrangements, J. Bonnett, Monsanto Canada Limited.

# Industrial Literature

## AMF Booklet

A 56-page booklet on STANDARD AND SIMPLIFIED DRAFTING PRACTICES is being offered free by American Machine & Foundry Company. The first section of this 8x11½-inch publication contains a speech on this provocative subject as given by Jay H. Bergen, AMF Standards Administrator, before the American Society for Engineering Education. The remaining 44 pages are devoted to the details of AMF standards on Drafting Room Practice and Simplified Drafting.

These 44 pages on traditional and simplified drafting procedures show how AMF has put into practice author Bergen's "supplement" philosophy. The 20 pages devoted to Standard Drafting Room Practice, as recently revised cover in a comprehensive yet simple manner all basic drafting operations and then wind up with 4 pages of helpful data on general procedures.

The final 24-page section on Simplified Drafting starts out with the 11 rules as covered in detail by Mr. Bergen in his widely circulated booklet, SIMPLIFIED DRAFTING, and then goes into the specifics of simplified practice. To expedite cross reference between specific items in the traditional and simplified standards, similar numbers have been used for similar paragraphs.

Those interested in obtaining a free copy of the new 56-page booklet may write on company letterhead to: Public Relations Department, American Machine & Foundry Company, 261 Madison Avenue, New York 16, N.Y.

## New Rod Sizes

New extrusion equipment developed by Tri-Point Manufacturing, Inc., Brooklyn, N. Y., specialists in extruding Teflon, now makes possible advantageous changes in sizes and prices of precision-extruded Teflon rod supplied by the Company. According to the schedule issued today, lengths and choice of diameters are increased, which will cut sharply waste experienced in machining of parts from "oversize" extruded stock.

Further information and copies of the new extruded Teflon rod price schedule are available from Mr. Pat Ruggieri, Tri-Point Manufacturing, Inc., 401 Grand Street, Brooklyn 11, N.Y.

## American Alkyd

The American Alkyd Industries announces it is in production on a complete line of polyester resins. In addition to its Amester 722, general purpose; and Amester 722 L.S., light stabilized; American Alkyd now has in production Amester 502, reactive; Amester 552, matched metal die resin; Amester 722 L.V., low viscosity; Amester 822, thixotropic; Amester 852, fire retardant; Amester 922, flexible and Amester 1022, gel coat.

Amester polyester resins for rigid and flexible foams, based on polyester-isocyanate formulations are available. American Alkyd Industries is in a position to supply these types or any type manufactured to the customer's formulation in any quantity.

For complete information send for free copy of our "Fact File on Polyesters," American Alkyd Industries, Broad and 14th Street, Carlstadt, New Jersey.

## House Organ Released

A new issue of its by-monthly external house organ, the "Plastics Weldor and Fabricator" has just been released by the American Agile Corporation, Maple Heights (Cleveland), Ohio.

The issue highlights an illustrated article entitled "New Horizons in Plastic: Containers," which describes various types of containers of plastic, their advantages and applications. Of particular interest is a chart which covers the relative chemical resistance of several container materials.

Another article illustrates and describes the company's newly introduced polyethylene pillows which are used to retard evaporation in open tanks and vessels as much as 70%.

Another in the regular series "Tips On The Hot Gas Method Of Gas Welding" is likewise included.

Copies of the free literature may be obtained from the American Agile Corporation, P. O. Box 168, Bedford, Ohio.

## Engineering Memo

An engineering memorandum on rigid unplasticized Polyvinyl Chloride pipe, fittings, and valves is available from the Peter A. Frasse and Co., Inc., 17 Grand Street, New York 13, N.Y.

## Pipe Dimensions

The Commodity Standards Division of the U. S. Department of Commerce has presented to the industry for consideration recommended Commercial Standards covering dimensions and tolerances for rigid polyvinyl chloride pipe, and solvent welded cellulose acetate butyrate pipe.

Both specifications were proposed by the Society of the Plastics Industry, and have been adjusted in accordance with comment from other interests. If they are endorsed by a satisfactory majority of the industry they will be issued by the Department of Commerce as Commercial Standards for voluntary use by the industry.

Copies may be obtained by addressing a request to F. W. Reynolds, Commodity Standards Division, U.S. Department of Commerce, Washington 25, D. C.

## Injection Machine Bulletin

A new eight page bulletin describes the operating features Watson-Stillman vertical injection molding machines which are available in 1, 2, 6, 16 and 24 ounce capacities.

Bulletin also describes the advantages of the vertical method of injection molding. Complete information on machine specifications and physical dimensions is also included.

Copies of Bulletin 627-B may be obtained by writing to the Watson-Stillman Press Division, Farrel-Birmingham Company, Inc., 109 Aldene Road, Roselle, New Jersey.

## Comet Industries

An 8-page booklet describing sheet plastic forming machines is available from Comet Industries, 9865 Franklin Ave., Franklin Park, Ill.

## Heating Booklet

A new 16-page catalog by Jensen Specialties, Inc. explains the latest techniques used in modern process heating, including a highly successful method of combining radiation with convection. Jensen points out that the catalog should be particularly informative to companies interested in a quick, easy-to-understand knowledge of modern heating systems.

The booklet, called "A Modern Advanced Approach to Process Heating" covers in detail, applications of booster ovens and complete systems, including information on panel segments, wiring channels and conveyors. A series of photo-stories show these parts in a variety of applications.

For your copy write Jensen Specialties, Inc., 9331 Freeland Avenue, Detroit 28, Michigan.

# NEWS of the industry

## Slide Rule Offer

The Du Pont Film Department is offering a handy new slide rule to designers and engineers with an invitation to compare the cost and properties of "Mylar" polyester film with those of other materials.

The introduction of this copyrighted cost and property comparator follows the recent announcement of the third price reduction of "Mylar" since commercial production started at the Circleville, Ohio, plant in 1954. Initial prices for this plastic film ranged from $3 to $4 per pound. Present prices are $2.25 per pound for all but the thinnest gauges.

Pocket size, the comparator works like a slide rule. The front side quickly determines cost per square foot of "Mylar" polyester film, compared to other materials. The reverse side matches the main physical, chemical, thermal and electrical properties of "Mylar" with those of polyethylene, acetate, acetatebutyrate, polystyrene, rag paper, varnished cambric, and polyvinyl chloride.

Designers and engineers may obtain a cost and property comparator by writing Du Pont Film Department, Wilmington 98, Delaware.

## New Casein

Development of biologically stable casein, resistant to microbial spoilage and insect infestation, is announced by the Chemical Division of The Borden Company.

Tests completed at Borden's casein development laboratory in Bainbridge, N.Y. show that preservatives commonly used with casein can be reduced or entirely eliminated in some formulas when Biologically Stable Casein is used. Samples of the product and complete technical data may be obtained by writing to The Borden Company, Chemical Division, Casein Department, 350 Madison Avenue, New York 17.

## Price Increase

A general price increase of one-and-one-half cents per pound for powdered shell molding resins used in the foundry industry has been announced by the Borden Company's Chemical Division. The increase, precipitated by rising production and delivery costs, is effective July 1. The price has been 26 cents per truckload pound and 27 cents per pound, less truckload.

## New Saw Available

A newly designed Traveling Cut-off Saw for rigid extrusion has been announced by The Rainville Company in Los Angeles. The manufacturer is The Vern-Emery Company.

This unit travels with the extrusion while cutting. The force of the extrusion is not used to move the carriage. The unit will cut up to 4" in diameter, and give a perfect, smooth, flat cut. It will make up to 30 cuts per minute. Several units are in use on pipe extrusion, including Polyethylene pipe, with excellent results.

The unit is distributed through The Rainville Company, 2415 West Hellman Avenue, Alhambra, California.

## Thiokol Sells Butyl

The Thiokol Chemical Corporation, Trenton, New Jersey, will market Butyl rubber, it was announced today. An extremely weather-resistant synthetic rubber, Butyl is used in the manufacture of inner tubes, tires, hose and a variety of household and industrial products.

Thiokol is entering the Butyl rubber field in collaboration with Petroleum Chemicals, Inc., a company jointly owned by the Cities Service Company and the Continental Oil Company, and presently operating a Butadiene plant at Lake Charles, La. Petroleum Chemicals, Inc., is a licensee under the Esso Research and Engineering Company for the manufacture of Butyl rubber.

## Coating Developed

General Plastics Corporation of Paterson, N. J., announced today the development of a new anti-static coating for industrial equipment, called GENCOTE 108.

The formula for the new coating utilized TEFLON as the base material, retaining all its characteristics but reversing the electrical insulation feature to create electrical conduction. Thus, it is now possible to achieve an anti-stick, dry-lubricated, chemically inert surface, that is anti-static as well.

## Vinyl Stabilizers

A new folder is now available which describes the line of vinyl stabilizers offered by the Nopco Chemical Company. A copy of the folder may be obtained by writing to Dept. VS, Metasap Chemical Company, Harrison, N.J.

## Men In The News

### New Appointments

Three new appointments in metropolitan sales offices of the Durez Plastics Division of Hooker Electrochemical Co. are announced by Robert E. Wilkin, vice president and director of sales. Lewis J. Pentland, Evan E. Graham, and Henry B. Puff have been promoted to district sales manager for New York, California, and Chicago respectively.

### Hanford Honored

Dr. William E. Hanford, director of research for the M. W. Kellogg Company, of New York City, designers and manufacturers of equipment for petroleum refineries, was one of three scientists to receive honorary degrees of Doctor of Science at the 135th Commencement of the Philadelphia College of Pharmacy and Science on June 11.

### Kimmel Appointed

Morris A. Kimmel has been appointed executive assistant to the general manager for the Resinous-Reslac Department of the Borden Company's Chemical Division. Long active in the adhesive compounding field, he has been with the operation, with headquarters in Peabody, Mass., since 1941. The executive assistant position is a new one.

### Chief Engineer

Arthur G. Ringlen has been appointed to the newly created position of chief engineer of J. E. Plastics Mfg. Corp., 555 West 23rd Street, New York City, manufacturers of acetate containers, it was announced today by Herbert Magnes, president. Mr. Ringlen will be in charge of converting the plant to automation, in line with the company's expansion program.

### New Appointment

The appointment of A. E. New as director of the technical department in the manufacturing division of Escambia Bay Chemical Corporation was recently announced by D. J. Stark, vice president and production manager. Mr. New has been director of process development at the Texas City plant of Carbide and Carbon Chemicals Company for a number of years.

### Transfer Announced

It has been announced by Mr. Vincent McCarthy, U.S. Industrial Chemicals Polyethylene Sales Manager, that Mr. J. K. Moffett, Jr., has been transferred to a sales development position for PETROTHENE(R) polyethylene resins.

# THE INJECTION molderscope

By A. R. Morse
*President, Injection Molder's Supply Co.*

## Polyliners Again

As manufacturing experience is gained on polyliners, it is increasingly apparent that they are far from the inexpensive item we have been led to believe. In manufacturing one for Stimsonite Division, for example, with some 480 holes, we used over 2 packs of #28 and #46 drills. Drills had to be resharpened every 5 to 6 holes. Drilling speeds had to cut way down to reduce the chance of drill breakage, especially on the #46 drills, as one broken drill would have scrapped the whole piece of fairly expensive 340 stainless steel forging. If it were possible to manufacture polyliners in volume, of course, gang drilling might reduce costs. Just for the record, the price of one polyliner insert 13½" long x 4½" in diameter, with approximately 480 holes, drilled, reamed, and polished, with 22 slots or grooves .016" deep will run about $1650.00 with the price varying 10% one way or the other, depending on just how much hand polishing is needed to take out tool marks. Note that this is the cost of the polyliner **insert** only, and the heater barrel or body would be extra.

In another instance, we made one polyliner cylinder on an order calling for three. After their first was delivered, and tried, the balance of the order was cancelled; and we could not get anything more than a grunt when we inquired why. What we want now is some **positive evidence** that the polyliner is all it was initially reported to be. We hope to be able to persuade either DuPont or Dow to give us a feature column on it before too long. Maybe they are getting some production figures that will substantiate the underlying theory. That the melt extraction, (Dow), or polyliner, (DuPont), theory, has real merit cannot be denied. Here is a letter outlining how Bob McKee of Dow feels about my previous comments on it:—

Dear Sir:

I was happy to see your comments on the melt extraction (Maccaferri, or Polyliner) type of heating chamber printed in the Journal, where they may be commented on.

Of course, it is true that most basic progress is a slow, halting thing. But that does not make slowness a virtue, and innovation a sin! We'll do the industry, and ourselves, a service by evaluating this new design type on its merits alone.

It is true this year, as it has always been, that machine tools of quality and performance are not cheap. I don't believe anyone has tried, as you infer, to give the impression that these units are "easy and cheap". They can be a sound, money making investment for the molder who can use extra performance.

Our design system for the merit extraction type heater is just an engineering approach to a fairly common engineering problem—the design of a heat exchanger. We assume that each injection "skims off" from the heated primary surface a volume of plastic equal to the shot (hence the name melt extraction). If the shot size and the primary surface are known, we can 'arithmetic' the thickness to be melted each cycle. The time required to heat this thickness of plastic is the minimum cycle for the shot size in question. You can play with L/D ratio, slot width, slot depth, hole size, cold spreaders, and ouija boards till doomsday. If you don't have enough surface, you won't get the production you need.

You have stated, many times, that the range of these designs is limited ("the . . . cylinder that will run a one ounce job on a 12 second cycle does not appear to be able to run a 4 ounce job . . ."). I submit that a molder should not expect a one ounce cylinder to shoot 4 ounces. I am sure you wouldn't approve of anyone trying to get 80 gallons from a 20 gallon pump. Nor would any engineer try to get 400 tons of clamp from a 175 ton machine. For best results, the machine is matched to the job. For a 4 ounce shot, use a 4 ounce heater. Isn't that ordinary common sense?

The cycle to be run is an important factor in sizing these heaters, because a thin section will heat so much faster than a thick one. For that reason, while a conventional heater is good for so many pounds per hour, the melt extraction heater will actually produce many more lbs./hr. at a fast cycle and small shot. To be sure, the designer should calculate primary surface area for the largest shot he is likely to run and the fastest cycle that it will be possible to run that big shot. (As determined from mold closed time, etc.) Then this decision can be checked against other likely shot-cycle combinations. The calculation is explained in an article in the February issue of SPE Journal. Copies and advice are available from Gordon Thayer, Plastics Technical Service, The Dow Chemical Company, Midland, Michigan.

The question of overheating on smaller-than-design shots has been raised. In our favor is the fact that a slight decrease in temperature (6°C) will about halve the rate of decomposition. In practice, you will find that reduced heat and reduced injection pressure are in order when running a Maccaferri cylinder below its rated shot or cycle. We (Dow Chemical) don't know of anyone having burning problems with these units.

I hope the foregoing will reduce your objections to the design system. As you say, the wide range of size and specification in molding machines makes a heater design based on rules of thumb pretty chancy. In the application of heat transfer principles and engineering common sense to the development of these heaters, we can earn our keep—and like it.

Very truly yours,

**R. B. McKee**

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**The Injection Molderscope solicits articles on any phase of injection molding from anyone who has ideas he wants aired. We want stimulating articles and comments from anyone that can be adapted for publication. So let us hear from you — on machinery, on powders, on molds — it is your chance to be heard.**

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# AROUND THE WORLD

## ARGENTINA

REVISTA de PLASTICOS

March-April, 1956

Abstracter: John B. Ballard

**BEGINNING IN SEPTEMBER, MONSANTO ARGENTINA WILL PRODUCE POLYSTYRENE — Interview with Ing. Enrique E. Krag.**

Monsanto Argentina is building a new plant near Zarate, Buenos Aires Province, which will be producing polystyrene about September, 1956, and, by early 1957, will be producing several types of polystyrene including high-impact. The basic raw material, styrene monomer, will be imported.

## BRAZIL

REVISTA BRASILEIRA DE PLASTICOS

June, 1955

Abstracter: Louis A. Helwich

**PETROLEUM, THE GROWING INDUSTRY OF BRAZIL. Page 4 — 7.**

A report of growing activity in the petroleum field in Brazil and discussion of potentialities of producing plastics materials by way of petrochemicals. The Bahia oil fields count now 251 wells producing oil and 30 producing gas. The refineries in Mataripe and Cubatao are described. New refineries are planned in Porto Alegre, Curitiba, Federal District, Recife and Belem. Also deposits of asphalt and bituminous schist are being exploited.

**SILICONES IN THE INDUSTRY — E. A. Running. Page 8.**

A brief explanation of chemistry of silicones and their principal applications in treatment of textiles, as electrical insulations, lubricants, rubbers, parting agents.

**PLASTICS IN THE WORLD Page 16-17.**

Some news is reviewed: Polyurethane foam; Increased production of polyester film Mylar; Polyethylene-covered paper containers for foodstuffs etc.

**INJECTION MOLDING—NEW THEORIES ON INJECTION — Eric Jeanrenaud. Page 18 — 21.**

Low pressure injection molding, a procedure much discussed in Europe and USA is given an extensive treatment, based on experiences in France. The principle is to attain a maximum of fluidity and homogeneity of the material after leaving the cylinder up to the end of its run in the mold. The pressures are of the magnitude of 300 kg/cm² as compared with the regular high pressure technique using pressure level of about 1,500 kg/cm².

**BRAZILIAN PLASTICS INDUSTRY CALLS FOR MOLDS — Page 22 — 23.**

The plastics market is already sufficiently large in Brazil to justify establishment of tool industry on large scale. The present volume of mold production is insufficient. Originally Sao Paulo was the center of plastics activity, however, now other states are beginning to take part in it and new plants are being built.

July, 1955

**BAKOL S. A. A PROGRESSIVE COMPANY IN PLASTICS — Page 4 — 8.**

A description of polystyrene plant of the company in Sao Paulo district. Methods of polymerization of styrene are reviewed and the process employed by the company, which is said to be without catalyst, is described.

**PACKAGING — A VAST FIELD OF APPLICATION OF PLASTICS — Page 11 — 14.**

A general discussion of the present situation. Applications of plastics as containers, jars, bottles, film and foil etc. are listed. Growing interest of the Brazilian public is noted.

**INJECTION MOLDING—NEW THEORIES ON INJECTION — Eric Jeanrenaud—Page 25—29.**

Theoretical treatment of thermal requirements of various materials for optimum injection process.

August, 1955

**PLASTICS IN SERVICE OF ADVERTISING — Page 4 — 7.**

A review of numerous applications of plastics in this field. Methacrylates, acetate, vinyls, polystyrene, polyethylene, reinforced plastics are discussed and examples from Europe and the USA given.

**NOVOLIT S. A. — THIRTEEN YEARS OF SUCCESSFUL ACTIVITY — Page 10 — 14.**

A description of a modern molding plant in Sao Paulo.

**INJECTION MOLDING—NEW THEORIES ON INJECTION — Eric Jeanrenaud—Page 19—22.**

A summary of operating conditions for low pressure injection molding process: Adjustments and adaptations of the mold required.

**BRAZIL INTERESTED IN ITALIAN PLASTICS MACHINERY — Page 25 — 29.**

Two Italian firms are introduced to the Brazilian market as suppliers of processing machinery e. g. injection molding machines, extruders and, an innovation in Brazil, vacuum forming machines. (Negri Bossi & Cia. in Milan and Officine A. Triulzi). Technical description and illustrations of the machines.

## GERMANY

KUNSTSTOFFE

May, 1956

Abstracter: Anne Schwend

**LOW PRESSURE POLYETHEYLENE PIPING — K. Richard and G. Diedrich**

This survey on the suitability of low pressure polyethylene is concerned with showing how far such tubing

can already substitute metal pipes. Good chemical resistance against all sorts of acids and alkalis with the exception of concentrated oxidizing acids and halogens, low swelling from solvents, the possibility of laying great lengths of light weight tubes from drums on which they may be coiled, easy fitting and their insenitiveness against bending stresses as well as the absorption of compressive or stretching forces make them well suited for laying on or under plaster or in the open air where frost damages occur less than with metal pipe lines. Extensive investigations are under way to gain experience from practical application.

### USE OF FOAMS FOR ACOUSTICAL PURPOSES—H. Oberst

The low density of soft foamed plastics, their great dynamic resilience coupled with low velocity of sound, their excellent compressibility as well as low resistance against volume modifications make soft high polymer foams with through-pores or closed cells most suitable for sound deadening or the attenuation of bending vibrations of droning metal structures, especially in cases where sound deadening layers cannot be sprayed on or knife-coated or good heat insulation is required in addition. Not only can PVC be laminated with a soft rubber layer to thus shift resonance to lower frequencies by making use of the modulus of elasticity of the cover material, but the field of mechanical dispersion inherent to cellular PVC can also be alligned to the frequency range of interest by the means described. Almost complete sound absorption within relatively low audio frequencies as well as above the lowest thickness resonance frequency are obtainable by cited techniques.

### CHEMICAL AND PHYSICAL PROBLEMS UPON MANUFACTURE AND FORMING OF THERMOPLASTIC PLATES AND FILMS — H. Moritz and R. Ewald

Co-polymers of PVC with vinyl acetate represent a good product for vacuum forming and plates from rubber-modified polystyrene have so far met even extreme requirements. In order to get closer to gauging thermoplastic forming more accurately, torsional tests were conducted and revealed that the torsion modulus G depending on temperature and the measurable damping are an excellent criterion for gauging the structure of high polymers from which the properties, held essential for vacuum forming, have to be derived. Although three-dimensionality is a characteristic of vacuum forming, also tensile tests in dependence from time yield a good criterion for the formability of plates. In any case, the temperature of the tension maximum is a function of the forming speed. The possible degrees of forming above the tension maximum increase with the forming speed.

Chemical aspects are only briefly touched: absolute uniformity of materials is underlined, choice and amount of lubricants, plasticizing influence of stabilizers, varying pre-heating periods upon use of fillers and colors, influence of different degree of acetylation.

### PVC PASTES WITH IMPROVED PROPERTIES — G. Wick and G. Weber

In order to bring processors up to date on the production and application possibilities of technical and special PVC pastes, this resume acquaints them with variable property details, homogenization, de-aeration under vacuum, use of extenders, thinners and fillers with stabilizers for cold dipping.

### PLASTICS REACTION UPON INFLUENCE OF SHEARING STRESSES — E. B. Sharp and B. Maxwell

Excerpt from Modern Plastics, December 1955.

### HYDRAULIC DRIVES FOR THERMOPLASTIC INJECTION MOLDING MACHINES — A. Blumer

A juxtaposition of demands as to pressure regulation and wide-range adjustability with the possibilities offered by pneumatic, mechanical and hydraulical transmission of engine power decides in favor of hydraulic drives for injection molding machines. Completely washer-less pumps, control members and values which reduce the up-keep of an oil hydraulic injection molding machine to a minimum, simultaneous lubrication of practically all movable parts by the hydraulic oil and the advantage of completely vibration-free oil flow give oil hydraulic drives preference over water hydraulic drives. The possibility of intercalating an accumulator is stressed.

### CATERPILLAR TAKE-UP DEVICE FOR PLASTIC TUBING

Description of a new take-up device for plastic tubes. After having run through the calibrator the still warm and deformable tubes are seized between an upper and lower caterpillar chain the speeds of which are infinitely variable and absolutely uniform. This device is manufactured by Maschinenfabrik Johann Leimbach K. G. at Wuppertal-Elberfeld.

### EXTRUSION DIES — G. Schenkel

Part II of this article holds descriptions of sheet blowing dies, flat type sheet dies with "Manifold", fan-tail and wide slot dies, dies for extrusion laminating for coating of paper, textiles, aluminum, etc; further, dies for multi-colored or reinforced extruded profiles and tubes, multi-colored spiral coated wires and dies for intermittent operation for extruders having an injection molding or mold blowing machine attached.

### DESIGN OF INJECTION MOLDS — Hans Gastrow

Injection molds for parts with internal threads or hinges are discussed.

### FEED WEIGHING FOR INJECTION MOLDING MACHINES

An excerpt from Dow Digest No. 18, completed by descriptions of feed timers of German origin.

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## GREAT BRITAIN

PLASTICS

March, 1956

Abstracter: Alfred T. Rexer

### MODERN METHODS OF MOLD MAKING—Part II. Copy Milling and Turning. Page 90

This is part II of a series and describes copy milling and turning. The machinery illustrated and described is more or less conventional and includes universal milling machines, pantographs and hydraulic or electronic links.

### CONTINUOUS PRODUCTION OF VINYL FOAM—Page 86-88

This article is a description of an American method patented as early as 1952 by the Elastomer Chemical Corporation of Newark, New Jersey. This method did not use so-called chemical blowing agents, but uses absorbed gases as carbon dioxide. A flow sheet for the continuous production of vinyl foam is discussed. The actual cure is performed by dielectric "high frequency" heating. The curing temperatures run about 148°C for a 2" thick sheet moving at the speed of 18" per minute giving a total production of 400 lbs. per hour. Various densities can be made ranging from 3.5 to a 30 lbs. per cubic foot but the standard density is 5 to 7 lbs. per cubic foot.

# SPEAKING OF EXTRUSION

*Edited by* R. D. Sackett
Technical Service Associate
Monsanto Chemical Company
Springfield, Massachusetts

## *Extrusion Dies For Plastic Pipe*

R. S. Perkins

*Southwestern Plastic Pipe Co.*

Interest in plastics extrusion has reached an all time high, prompting a great deal of research and investigation with the net result that techniques of plastics extrusion are rapidly progressing from what once had been referred to as an "art" to one that is based on scientific fact and proven theory.

A tremendous amount of work has been accomplished particularly in regard to screw design and machine performance for various types of materials. These developments, of course, have been of great benefit to processors. The processor is interested primarily in high poundage output of an extruder together with a "surge," or pulsation free delivery of a completely plasticized stock. We recognize that all materials exhibit different extrusion characteristics, and many specialized techniques can be developed for different operations such as nylon extrusion, polyethylene extrusion, coloring operations, vinyl dry blend, etc. Few of us are equipped, however, to set up individual machines for one specific type of material or one specific operation, so we must have equipment which is versatile enough to process all types of materials with a minimum of changes and modification.

In spite of all development in equipment, and the improvement in machine efficiency, very little work has been published to date concerning effective methods to produce extruded shapes at rates embodying the maximum machine capacity.

Let us take pipe extrusion for example, and more specifically, the die design necessary to produce quality products at acceptable rates of production. Plastic pipe and tubing are simple geometric shapes and appear to be the easiest shapes to extrude. Strangely enough they can create many production problems. Admittedly, die construction for tubular products is relatively simple and straight forward, and many rules of thumb have been advanced concerning correct land lengths for various sizes and wall thicknesses of tubular products. The most common rule being "a land length equal to the O.D. of the pipe or tube." This particular rule of thumb leaves much to be desired so we attempted to develop some sort of a criterion for determining land length for tubular goods for varying wall thicknesses as well as for various types of materials. The system we devised has worked out extremely well and was very advantageous in eliminating much of the die development work previously encountered in establishing production items.

Recognizing that in pipe manufacture, wall thicknesses in any one particular pipe O.D. can run from .065" to .225", some consideration must be given to the back pressure developed at the die by the parallel land length, and the orifice thickness.

If land lengths are too short, insufficient fusing of the material will take place particularly where the flow is broken up by torpedoes or spiders. These flow lines or zones of incomplete fusion will result in pipe of lowered burst strength. On the other hand, if the land lengths are too long, excessive back pressure is built up and output or productivity is apt to be decreased and, too, there is the danger of placing an excessive amount of strain on die holders and gates.

Recognizing these factors, an experimental program was initiated to determine optimum land lengths for the various materials as well as develop some sort of a mathematical relationship between annulus and land length, as it stands to reason that for tubing with heavy wall, even though the O.D. may be quite small, the longer the land length must be in order to effect good caliper control and maximum pipe strength. First of all it was determined that with polyethylene and with Tenite Butyrate, for example, a much longer land length was required than for either Kralastic or rigid PVC. The reason being that the more fluid the material, the longer the parallel land must be in order to maintain good size and thickness control, whereas with stiffer materials, a much shorter land is required. It was first thought that a direct ratio of land length to annulus would be the answer to producing quality pipe, but continued experimentations indicated that this particular relationship would not hold true for various sizes and wall thicknesses of pipe. Several other approaches were tried and it was finally determined and proven on production dies that the following formula was most applicable to the rigid materials such as Kralastic, PVC, etc. This formula reads

Land length (inches) = (annulus x $\pi$) f

f = 10 for PVC & Kralastic

f = 15 for polyethylene & Tenite Butyrate

This particular approach resulted in excellent performance irregardless of pipe size or wall thickness.

No decrease in output was noted and the products produced exhibited improved properties over the same products extruded under less favorable conditions.

## St. Louis Meets

Parke Woodworth

The seasons final meeting of the St. Louis Section was held at the Melbourne Hotel on June 19. Reviewing a custom that had been dormant for some time, the St. Louis Section invited wives and sweethearts to participate in the summer meeting. The feminine contingent accepted with enthusiasm and added significantly to the enjoyment of the session.

Dr. Norman A. Lewis, project specialist with Monsanto's Organic Chemical Department tempered his remarks to give the ladies an understanding of the complex nature of plastics. He reviewed the origin and manufacture of polymer in terms of their molecular structure. By outlining relationship between polymer molecule, he was able to explain some of the differences in how plastics behave in processors plants and end use applications. This was followed by Monsanto's outstanding film, "The World That Nature Forgot," visually describing the manufacture and utilization of plastic materials.

The next meeting of the St. Louis Section will be Tuesday evening, September 18.

# SECTION NEWS

*Edited by*

**Stanley Bindman**

*Jamison Plastic Corp.*

*1255 Newbridge Road*

*North Bellmore, L. I., N. Y.*

## Southern

## *Polyethylene Molecule Research*

John W. McConley

The polyethylene molecule and its effect on processing and end-use performance characteristics has been the recent subject of intensive study by du Pont research chemists. A report on these findings was the background of a talk by Dick Hardesty at the June meeting of the Southern Chapter. Members also saw a sound-movie on the "Polyliner", a new development by du Pont to improve efficiency of injection molding machine cylinder design.

Melt index density and molecular weight distribution are three distinctly separate measurements and indicators of a polyethylene molecule's performance. The MWD, as he referred to the distribution measurement, is the least understood of the three tests and is a general indicator of the frequency of the weaker links in the chains. All three are valid and independent measurements of characteristics, however, and all should be recognized in selecting a polythylene for a specific end use.

Density, or degree of crystallinity, is quite similar in its effect on properties as with metals. Increases in density cause increases in stiffness, yield strength, upper softening temperature and resistance to creep. Melt index is a measure of function flow of the molten polymer and is of function molecule size, distribution and chain branching. It is primarily an indicator of fabricability, but is useful also for selection of polyethylenes with better resistance to environmental stress cracking. The higher figures of melt index indicate better flow or greater number of smaller molecules, so when the properties of the larger molecules are desired, such as stress-crack resistance, the melt indexes around 1 are to be selected.

The range of molecular weight distribution for common commercial polyethylenes is from 3 to 60 with the narrow range being at the bottom end of the scale. Narrowing distribution, or increasing uniformity of molecule size, results in increases in tensile strength, resistance to stress cracking and ease of processing at high shear stresses.

These methods of measurement are found to have the same importance in characterizing the polyethylene of both high and low pressure processes.

The "Polyliner" is a bored and fluted liner to replace the torpedo in most standard injection molding machines. The recommendations based on du Pont's development work have been made available to machine and accessory manufacturers and "Polyliners" may be procured through these normal channels.

The Southern Section has suspended meetings for the summer season and will resume its regular schedule on September 17.

## Upper Midwest

## Plastics Fair Held

F. W. Brown

The Upper Midwest Section closed this season's activities with a Plastic Fair and dinner held May 21 at the Prom Ballroom in St. Paul. Response to the fair greatly exceeded our expectations with 1400 people viewing displays set up by the forty companies participating. The displays showed the wide range of products produced and the importance of the plastic companies in the Upper Midwest area. The Upper Midwest Section is the first section of the S.P.E. to hold such a fair.

A brief business meeting and dinner followed the fair. Mr. Cort Platt chaired the meeting. Mr. Jerome Formo, our national president, reviewed the rapid growth of the S.P.E. and set a goal of doubling S.P.E. membership by 1959 giving a national total of 10,000 members. Mr. Formo also asked for support of the Upper Midwest Isocyanate Symposium scheduled for October 23.

Mr. Paul Felt acted as general chairman of the fair committees and gave thanks to the committee chairmen and the many persons whose work made the fair a success. The section also gives thanks to Mr. Felt for his hard work.

After the meeting dinner was served to 287 S.P.E. members, wives, and friends. The dinner speaker was Mr. Brooks Stevens, noted industrial designer. Mr. Stevens gave us a glimpse into the future as seen by an artist and showed us what style trends are expected in the automotive and in other industries. He traced the history of industrial design and showed the impact of the industrial designer in past years in making new inventions such as sewing machines, trains, and automobiles acceptable to the public. He explained the theory of "planned obsolescence" and showed that it does not lead to waste as is sometimes assumed. Mr. Stevens illustrated his talk with many colored slides. His talk was informative as well as very interesting.

## *New York*

## Two-Part Program

Jim Dugan

SPE's New York Section featured a two-part program at its June 20 meeting. The Gotham Hotel in New York City was the site, and approximately 70 members and guests were in attendance.

A film entitled "Color Dynamics in Industry" was shown by A. Cerniglio, Pittsburgh Plate Glass Co., following a few introductory remarks. The film demonstrated the effect of color, and combinations of color, on the human eye, and how the proper use of color in a plant reduces eye-strain and absenteeism. According to established codes, certain colors are used to indicate fire apparatus, disposal and refuse areas, dangerous chemicals, and duct systems.

"Factors Affecting the Properties of Polyethylene" were pointed out by Richard H. Hardesty, E. I. du Pont de Nemours & Co., Inc., particularly with reference to fundamental molecular characteristics. The five most important factors were presented as follows: weight-average molecular weight, number-average molecular weight, molecular weight distribution, number of short-chain branches, and the long-chain branching index. The various properties were defined; stiffness, yield strength, tensile strength, tear strength, permeability, processibility, film brittleness, low-temperature brittleness, Vicat softening temperature, flex life, creep, and environmental stress-cracking; and it was brought-out how these properties are affected by the aforementioned molecular characteristics.

## *N.Y. Reinforced*

# Fire, Dermatitis Precautions Listed

Jim Dugan

"Safety in the Reinforced Plastic Shop" and "Synthetic Fibers for Reinforced Plastics" were the two topics under discussion at the June 27 meeting of SPE's New York Section, Reinforced Plastics Group. About 30 members and guests were in attendance at this, the last meeting until Fall.

J. Robert McCullough, Budd Co., opened the discussions, describing how his firm handles the problems of fire and dermatitis encountered in dealing with polyester and epoxy resins. Among the precautions followed by Budd are the following: having only a single-day's supply of material on hand in the working shop; the use of an explosion-proof room for mixing; use of large hoods to draw off over-spray; no body contact with the material; and special clothing and equipment. Special protective creams, such as Curadex, are used in quantity by the epoxy handlers, and workers are removed from the area immediately on sensitivity.

Nylon, Dacron, and Orlon fibers were described as polyester reinforcements by DuPont's John G. Green. Fibers were shown in three forms: short, trimmed fibers; continuous, rope-like tows; and mechanically-bonded, non-woven mats. Abrasion resistance with synthetic-fiber laminates was reportedly good, as is the less than 1% water absorption. In addition, Orlon and Dacron laminates show little or no light degradation in tests covering almost two years. Some of the most likely advantages of these laminates are thought to be chemically-resistant overlays in ducts and tanks, and epoxy filter frames. Care should be taken in fabrication, however, since hydrophobic fabrics tend toward poor adhesion.

"Approaches to Molding Complex Parts" was the problem under discussion at the May 23 meeting of the Reinforced Plastics Group, New York Section of the Society of Plastics Engineers, Inc. Approximately 40 members and guests attended the meeting, which was held at the Gotham Hotel in New York City.

Melvin Rubin, Steiner Plastics, discussed "Toy Car Body Manufacture," covering the problems encountered in making the Ford Thunderbird, Jr. Ford furnished the original prototype, and Steiner produced 2,500 units in the past two years. Basic considerations were as follows: should the item be molded in one piece, or as several units?; what material should be used?; and what methods could be best employed? The model car was molded of reinforced plastic materials by the air-diaphragm method, using 12 psi. pressure for 1½ hours. Because of complex undercuts, a split mold was necessary; however, as many as 60 units were produced per week.

"Molding Complex Parts by Means of Contact Pressure" was the subject of a talk by Irving Einhorn, Budd Co. The specific item under discussion was a metal rail car with a reinforced-plastic interior. The problem involved the proper choice of resin-catalyst system. The materials were always checked in the mixing room for proper cure, contamination, viscosity, and color. For the latter, Budd found DuPont's colormeter or colorimeter to be most effective in color matching, particularly when long intervals elapsed between runs.

Celanese Corp. of America's Michael Kallaur spoke on "Processes & Methods of Molding Complex Shapes from Premix Compounds," particularly with respect to getting reinforcements to follow complex curves. Material is cut to fill the contours, and a spray gun is used. Pretmix compounds are reported to be especially useful in the automotive field due to the cost differential. Lay up methods are not required, thereby eliminating the necessity of frequent hand-washings. Flexibility is another important consideration.

### May Meeting

A two-part program was featured at the May 16 meeting of SPE's New York Section, held at the Gotham Hotel in New York City. Approximately 80 members and guests were in attendance.

"Basic Packaging, Handling, and Loading Considerations" were covered by C. M. Bonnell, publisher of "Pack aging and Shipping". The primary purpose of any package should be its ability to deliver a product to the ultimate consumer in a useable condition. Concerning terminology, some firms and the government refer to interior containers and wrappers as packaging, and designate the exterior or shipping container as packing. Interior packing has advanced from the three old stand-bys of hay, straw, and excelsor to imbedment in plastic foams. Mr. Bonnell also described the following types of shipping containers in some detail: nailed wooden boxes, fibre containers, solid fibre board, corrugated fibre board, plywood boxes, crates, wirebound crates, wooden barrels, metal drums and pails, bags, and pallets or skids.

"Auxiliary Mechanisms in Automatic Molds" were the subjects under discussion by Milton A. Sanders, Consultant. Considerable attention was devoted to automatic cams and to the various unscrewing mechanisms which have been employed. It was brought out that in many instances the mold maker does not understand the problems involved in molding, and vice versa. Mr. Sanders also answered a number of questions directed from the floor, largely concerned with mold temperatures and mold closures.

## *Newark*

# 150 At Field Day

R. Bostwick

The second annual Field Day of the Newark Section was held June 7 at Mazdabrook Farms, Parsippany, N. Y. About 150 people turned out for the all-day affair of lunch and banquet dinner, along with assorted athletic activities and numerous door prizes.

Best summer wishes to all our friends in the Newark Section. We look forward to another series of enjoyable and informative meetings in the Fall (our Program Committee is already hard at work).

## Frankly SPEaking

Last month, for the first time, you saw this column. We hope that you read it and wondered how it happened to appear in the SPE Journal. Well—this month is devoted to a statement of what I hope to accomplish by writing a more or less integrated series of columns on various and sundry subjects. Actually, this is only a continuation and expansion of the open letters that I have been writing to you for the last five years. I hope you will consider each column as a personal letter to each of you.

I hope to accomplish at least one of several things with each of these columns. FIRST: If someone deserves a pat on the back,—they shall receive it. SECOND: If the pat should be a little lower down and administered with the foot, it is quite likely that is the way it will happen. THIRD: Maybe I can remind you of something you had forgotten. FOURTH: I expect to be highly critical of some of the foolish, selfish or dangerous ideas that are being pushed upon us as "truth". FIFTH: I should like to feel that one of these columns will be an inspiration to some one of you to make the decision to do the "right" thing. SIXTH: We call ourselves a Christian nation. Can you honestly say that you remember this when you have an important decision to make?

In closing, I hasten to say that I do not pretend to be able to live up to all the high ideals that are a part of our Christian heritage. I only say that we need not be pertect to talk about perfection any more than we need to be rich in order to talk about money. Perhaps the best summation of what you can expect here is to say that it will be a mixture of praise, inspiration, criticism, discussion and just plain personal opinion.

Wayne I. Pribble

## Reinforced Plastic Springs . . .

(Continued from page 14)

before curing.

Although the springs thus far described have been designed to meet the requirements listed in Section II it is obvious that the properties of plastic springs can be widely varied. For example, stiffer and probably more brittle springs result when the glass content is increased. The number of coils per unit length may be decreased by using tubing of greater wall thickness and dimensions may be varied as required by other applications. Where the temperature range is limited, other resins such as the polyesters may be economically substituted.

Table 6. Composition of Some Glass-Epoxy Plastic Springs

| NBS Spring Number | Resin | Glass Content[a] % | Number of Double Rovings[d] | Wire Diameter In. |
|---|---|---|---|---|
| - | 28 | 66 | 5 | 5/16 |
| 7 | 28 | 61 | 7 | 1/4 |
| - | 28 | 66[b] | 9 | 1/4 |
| 11 | 28 | 64[c] | 9 braided | 1/4 |
| 5 | 28 | 47 | 6 | 5/16 |
| - | 28 | 53 | 8 | 5/16 |

a. Average of three specimens.

b. One specimen.

c. Average of two specimens

d. A glass roving, doubled and drawn through the form used. The roving has 60 strands of glass fiber.

### Summary

Helical plastic springs with coil diameters of approximately 1-inch and wire diameters of 3/16-inch were formed from glass reinforced epoxy resins cured with *m*-phenylenediamine. A spring of this type, two inches long deflected ½-inch under a static load of 25 lb. Springs 3-in. long, compressed to their solid length and stored at 135°F for 13 days, retained over 5-in.-lb. of energy (43% of original) at the end of the storage period. Substantial improvement in recoverable energy properties of springs was obtained by preloading before use. These glass-fiber reinforced plastic springs had torsional moduli of rigidity of the order of $1.0 \times 10^6$.

The springs were molded by drawing resin-soaked glass-fiber rovings through vinyl copolymer tubing and wrapping the loaded tubing in a helix around mandrels of suitable diameter. Mandrel and tubing were transferred to an air-circulating oven for curing. The tubing was subsequently removed.

A large number of commercial formulations were screened in order to eliminate resins with unsuitable properties. Tests of resin rods permitted a rapid evaluation of torsional rigidity and temperature sensitivity. In general, the polyesters were more sensitive to elevated temperatures than the epoxy resins, but the epoxy materials varied widely in this respect. Results obtained in this work and elsewhere indicate that polyester and epoxy resins are most promising for spring applications on the basis of torsional moduli and temperature sensitivity.

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## Adhesives in Aircraft . . .

(Continued from page 23)

prime reasons for this advancement are: (1) ease of application, (2) excellent basic adhering qualities to many kinds of metallic and non-metallic surfaces, (3) simplicity and versatility in compounding and development, (4) resistance to aircraft fluids and environmental conditions, and (5) satisfactory physical properties.

TABLE VI

Shear Strengths* At Room Temperature, 260°F, and 350°F Versus Concentration of Meta Phenylene Diamine

| Curing Agent (phr) | Room Temperature AV. | MAX. | MIN. | NO. OF SPECIMENS | 260°F AV. | MAX. | MIN. | NO. OF SPECIMENS | 350°F AV. | MAX. | MIN. | NO. OF SPECIMEN |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 13 | 2895 | 2960 | 2750 | 4 | 1105 | 1165 | 810 | 8 | 493 | 550 | 432 | 8 |
| 15 | 3100 | 3600 | 2685 | 20 | 3475 | 3750 | 2740 | 20 | 730 | 910 | 580 | 20 |
| 16 | 3380 | 4025 | 2640 | 8 | 3145 | 3810 | 2530 | 12 | 624 | 710 | 510 | 12 |
| 17 | 3424 | 3730 | 3140 | 8 | 2870 | 3450 | 2160 | 12 | 550 | 665 | 400 | 12 |

*All specimens bonded at 300°F, 100 psi, 30 minutes*

**psi (pounds per square inch)*

### Acknowledgment

The author gratefully acknowledges the many helpful suggestions given by H. P. Owen in preparing this paper. Acknowledgment is also given to K. E. Dorcas and Dr. N. H. Simpson for their review of this paper. All of these persons are members of Convair, Fort Worth, Engineering Test Laboratory.

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## Asbestos Reinforcements . . .

(Continued from page 27)

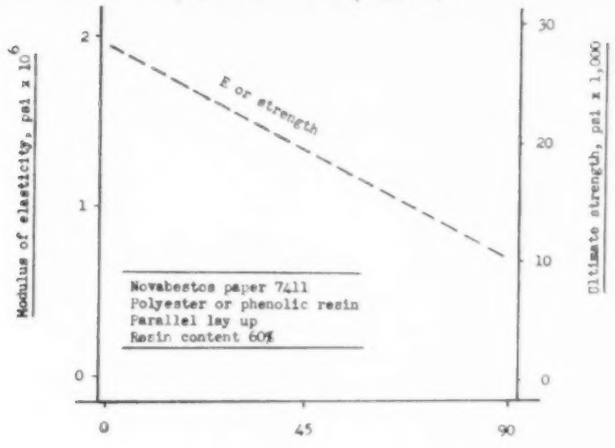


**Figure 8. Flexural directional properties of asbestos paper laminates**

Research and development continues to obtain more data in order to provide complete and concise information. Work to date shows that asbestos has definite fields of interests which include the following:

Creep and fatigue characteristics
Applicability of reducing hysteresis energy losses of glass-reinforced laminates
General chemical resistance
Applicability as adhesive carriers
Merits in tubular and rib structures
Dimensional stability
Electrical fields

Asbestos is definitely not a new engineering material; however, it is only recently that its usefulness in the reinforced plastic industry has been exploited. Designers, engineers and fabricators have begun to evaluate asbestos products with the aim of determining its suitability in specific applications ranging from nonstructural to highly stressed or structural items.

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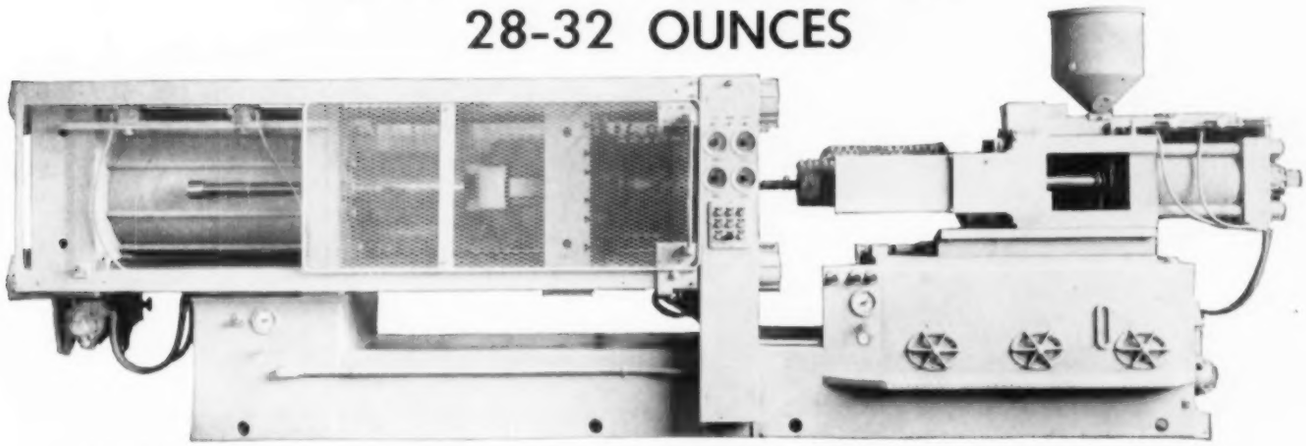

| | |
|---|---|
| Injection Capacity | 28-32 ounces |
| Injection Pressure | 21,200 p.s.i. |
| Plasticizing Capacity | 275 lbs. per hour |
| Injection Plunger Speed | 175" per minute |
| Clamp Pressure | Adjustable to 600 tons |
| Clamp Stroke | Adjustable to 30 inches |
| Machine Cycle | 250 per hour |
| Motor | 60 HP — 220/440V — 60C — 3 Ph |

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## Protective Potting . . .

(Continued from page 33)

and temperature cycling, also survived shock testing at an acceleration of 10,000 g's.

8. At accelerations of 14,500 to 18,500 g's, a significant number of tubes in each of the same groups failed. The percentages of failures at this level of acceleration were:

a. Uncoated tubes potted in epoxy resin, 44%.
b. Uncoated tubes potted in flexible polyester resin, 37%.
c. Silicone-rubber-coated tubes potted in epoxy resin, 14%.
d. Silicone-oil-coated tubes potted in epoxy polysulfide rubber copolymer, 35%.
e. Tubes in polyethylene resin-tight chambers potted in epoxy resin, 21%.

## Conclusions

General conclusions drawn from the above results may be summarized as follows:

1. Uncoated ceramic capacitors apparently can be potted directly in epoxy resin without fear of cracking or other failure.

2. Glass vacuum tubes should not be potted in epoxy resin without first applying to the tubes some kind of resilient coating. Silicone oil or a simple grease is not adequate. Silicone rubber, if applied in a thick enough layer, will probably prove satisfactory even against accelerative forces as great as 15,000 g's.

3. A polyethylene catacomb with resin-tight tube chambers affords protection by means of a compressible layer of air. In this case, additional protection against shock could probably be provided by looping the leads to lessen lead breakage and by further cushioning with fine glass fibers within the resin-tight chamber. Although this procedure may present an otherwise satisfactory solution to the problem of tube potting, it may be objectionable from a production-assembly viewpoint.

4. An epoxy polysulfide-rubber copolymer containing about 45 to 50 percent of polysulfide rubber appears to be sufficiently resilient at low temperatures to preclude tube breakage resulting from temperature changes. It is necessary, however, to coat the tubes with a release agent, such as silicone oil, before potting with this resin. Polysulfide rubber has an unpleasant odor and its use has, therefore, raised some objections. Furthermore, this type of copolymer fails to meet the dielectric requirements for a Type B resin as specified in Military Specification MIL-I-16923B and, therefore, it can not be considered for use in applications where electrical properties are critical.

5. The major objection to the wax-polyisobutylene mixture is its tendency to flow at temperatures approaching 160 F.

6. Although a larger amount of data would be required to allow a positive assertion to be made, it does appear that greater protection against mechanical shock is afforded a glass vacuum tube when it is enveloped in a resilient coating and then potted in a rigid resin, than is the case when the tube is potted directly in a resilient casting resin.

7. It is desirable to minimize shrinkage of the resin in a can containing an electronic assembly in order to: (1) prevent cracking of the resin, and (2) maintain a reasonable bond between the can and the resin. Shrinkage could be reduced by allowing the resin to gel at room temperature (time consuming) and by reducing the necessary volume of resin by designing a catacomb that occupied more of the space in the can. Stresses concentrate along sharp edges, and cracks in castings generally seem to start at these overstressed areas. Therefore, cracking of the resin could be reduced by the removal of sharp metal edges in the construction of the can.

## Acknowledgment

The authors desire to acknowledge the assistance of L. H. Bullis in establishing the relationship between pressure and filament current, and the assistance of Carl Rich and his staff in making the acceleration tests in the air gun.

## Low Temperature Shock . . .

(Continued from page 35)

on the surface, the temperature gradient and cooling curves can be found simultaneously.

In most units, there are several different materials having widely different expansivities. This of course precludes the matching of this property. The strength of the resin and modulus at varying temperature become critical, often the controlling factors. The shape of the unit may be such that excessive stress concentrations are built up at certain points. At present, complicated units must be cast in clear plastic and observed to determine the weak points. Often components will shield each other from observation.

Using the procedure outlined here, wire can be placed in several locations. From a single run much information can be obtained about the temperature distribution throughout a unit and positions most susceptible to cracking. This is particularly true in units operating at low temperatures where some components dissipate heat causing severe thermal gradients. From these experiments, more advantageous locations of these parts can be found. Often the conductivities of plastics can be increased with fillers to improve this situation.

The techniques of using painted conductive paths can be used where other methods are impractical. These paths can be applied to any electrically insulating surface without causing strain. There can be less than 1 microwatt heat dissipation if heating would interfere with the test. Glass vacuum tubes, plastic resistor cases and other such components can be painted and potted. An accurate record of the heating of these components can be recorded continuously and automatically. Temperature gradients across plastic castings can be obtained by casting the piece in layers and painting the surfaces of each layer with the conductive paint. Complex shapes and interfaces between differing materials can be analysed for heat conduction properties.

The wire wound units can serve a dual purpose. Temperatures during potting as well as those during thermal shocking can be recorded.

There has been considerable difficulty with reproducibility of epoxy casting compounds. Tests, such as epoxy equivalent, maximum temperature during setting, viscosity, and gel time, are used to check on consistency of batches.

In applications where cold shock resistance is of prime importance, the cold shock test could be used for quality control.

The technique of using wire wound forms in insert castings or painting a conductive path on the surface yields a test specimen that can be used for rapid, reproducible testing of cold fracture temperature. A recording potentiometer can be used to automate the test with little additional circuitry. The effect of fillers, plasticizers, curing agents, and resins can be tested. Shapes and sizes of embedments and plastic can be evaluated. The test can also be used for routine testing of resin.

# Book Reviews

**HANDBUCH DER NITROCELLULOSELACKE: LOESUNGSMITTEL.**
**(Handbook of Nitrocellulose Lacquers: SOLVENTS.)**
**by Dr. Alfred Kraus**
**Published 1955 by Wilhelm Pansegrau Verlag, Berlin-Wilmersdorf.**
**323 Pages.**

The author published the Second Part of his handbook in October 1952. It was to be followed later by the First Part, "Components of Lacquers," to include these 5 chapters: pyroxylin, plasticizers, resins, solvents, pigments. The wealth of available material, however, made it necessary to subdivide this part, the present outcome of which is the volume on "Solvents." It is a successful attempt to select the essentials from the copious literature on the subject, and to arrange them in easily accessible form. It contains valuable information for the chemist and chemical engineer who has a special interest in solvents.

After general definitions and classification of solvents, latent solvents, and diluents, this volume is again subdivided into 3 major chapters. Chapter 1 describes the properties of solvents from the point of view of their use in cellulose nitrate lacquers. Numerous tables and graphs cover such properties as evaporation rate, heat of vaporization, and lacquer flow; solvent ability, tolerance relations, cutting time, viscosity; solvent power and dilution ratio; moisture blush resistance; solvent retention; influence on mechanical film properties; compatibility with colorants; flash point, fire point, and autoignition point; solubility in water; toxicity; and odor.

Chapter 2, likewise illustrated with a great many tables, discusses the various groups of solvents themselves: esters, ketones, glycols, furans, nitrogen compounds, special mixtures, alcohols, ethers, hydrocarbons, halogenated compounds, and organic acids. The tables are not strictly confined to those solvents used for cellulose nitrate lacquers, but encompass the whole solvents field. The behavior of each technical solvent is thoroughly described, and the most important physical constants are listed. There are few theoretical considerations and no discussion of the manufacture of solvents.

Chapter 3 treats with the physical and chemical basis of the behavior of solvents in common compounds and especially in finished cellulose nitrate lacquers. Finally, the author develops his latest theories on nitrocellulose compounds and film forming.

Among the appendices is the listing of about 600 trade names of solvents, and a tabulation of 107 manufacturers of solvents, including 73 manufacturers in 9 countries other than the U.S. **Fred R. Homburger**


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Each Part is complete with a detailed subject index and a list of standards in numeric sequence. The 1955 Book of ASTM Standards, containing 2153 standards as compared to the 1898 standards in the 1952 edition, is about 13 per cent larger than the 1952 edition. Since 1952, 1158 standards are new or have been revised.

To keep the book up to date, supplements will be issued to each Part late in 1956 and late in 1957. As a service, a complete index is furnished without charge with each set of the book of ASTM Standards.

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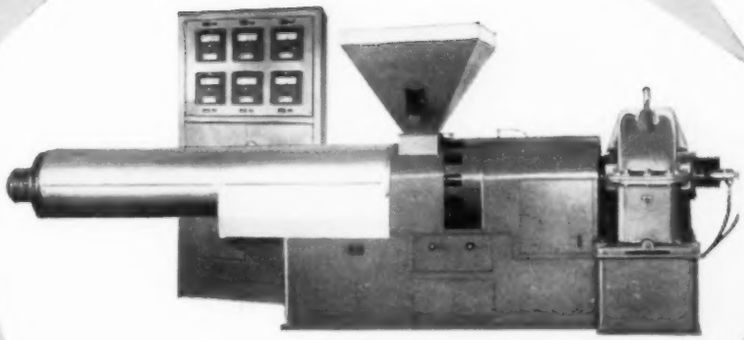

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